

INTERLABORATORY STUDY 89-2

**VALIDATION OF A METHOD FOR
RESIN AND FATTY ACIDS**

**SPIKED SAMPLES PREPARED IN
REAGENT WATER AND
PULP AND PAPER MILL EFFLUENTS
MARCH 1989**

JUNE 1990



Ontario

**Environment
Environnement**

Jim Bradley, Minister/ministre

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Report Prepared by:
Laboratory Services Branch
Ontario Ministry of the Environment

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PIBS 997
LOG 90-2743-002



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1 OVERVIEW OF THE INTERLABORATORY STUDIES TO ASSESS THE METHOD FOR RESIN AND FATTY ACIDS

In 1987, an Analytical Working Group (AWG) was formed to develop an improved analytical procedure for the analysis of Resin and Fatty Acids (MISA Group 26 and PP3). The AWG consisted of representatives from the Ontario Forest Industries Association (OFIA), Ontario Ministry of the Environment (MOE), and Environment Canada (EC), under the MISA program.

Initial work was done during 1987 to assess the stability of standards, the pH adjustments to be made prior to extraction, column conditions, and other instrumental conditions. The results have been reported elsewhere (3). In May 1988, the Method for Resin and Fatty Acids, Draft 3 (4) was ready to be validated by means of an interlaboratory collaborative study. A series of four separate studies were undertaken to assess the new method.

Interlaboratory Study 88-2A

This was the first study initiated to validate the performance of the Method for Resin and Fatty Acids, Draft 3 (4). Ampoules were distributed in June 1988. Four ampouled standards were provided to each participant, two to be spiked into reagent water and processed through the entire method, and two for direct methylation and injection into the analytical instrument.

The results indicated that the Draft Method produces similar results from all of the participants. Differences between laboratories may be attributed to differences in calibration of Dehydroabietic Acid. Some parameters are not recovered as well as others. This may be due to the difficulty of assuring the purity of the standards.

The results from Interlaboratory Study 88-2A demonstrated that the Draft Method works, as long as the calibration of Dehydroabietic Acid is carried out in a precise and accurate manner. The initial review of the raw data raised concern among the AWG regarding the use of two different solvents to prepare the standard solutions. A small follow-up study was required (see Interlaboratory Study 88-2B below), using fresh stock solutions, to assess whether some of the differences in performance were introduced by the different solvents used to prepare the standards.

Interlaboratory Study 88-2B

This was the second study to validate the performance of the Method for Resin and Fatty Acids, and was the follow-up to Interlaboratory Study 88-2A. Ampoules were distributed in August 1988. Four laboratories received two ampouled standards for direct methylation and instrumental injection. Each ampouled standard consisted of three parameters, one standard prepared in methanol and one standard prepared in methyl-t-butyl ether (MTBE). A third ampouled standard, consisting of the same three parameters dissolved in MTBE, but pre-methylated prior to sealing in the ampoules, was also provided to each laboratory. The third standard was to be used to verify that the four laboratories agreed in their calibration.

The results demonstrated that there was no significant difference between the mean of the results from standard prepared in methanol versus the mean of the results from the standard prepared in MTBE.

There was also no significant difference between the mean of the results from the standard prepared in MTBE that was not pre-methylated, versus the mean of the results from the standard prepared in MTBE that was pre-methylated.

Interlaboratory Study 88-3

This was the third interlaboratory study to validate the performance of the Method for Resin and Fatty Acids, Draft 3 (4). A set of ten samples was distributed to the participants in November 1988. The sample sets consisted of a High Spike, Low Spike, and Blank of Reagent Water, a High Spike, Low Spike, and Blank of Pulp and Paper Mill Effluent #1, a High Spike, Low Spike, and Blank of Pulp and Paper Mill Effluent #2, and one unspiked Pulp and Paper Mill Effluent #3.

The results indicated that the laboratories were able to recover the spiked Resin and Fatty Acids from the reagent water samples. Results from the two different spiked pulp and paper mill effluents produced very variable results, with very few laboratories reporting results from the low spike effluent samples. Reasonable consistency of results between laboratories was achieved for the unspiked Pulp and Paper Mill Effluent #3.

The variability of the results suggested that there may have been problems recovering the spiked Resin and Fatty Acids from the effluent samples. It was decided to repeat this study in the spring of 1989. This study was reported as Interlaboratory Study 89-2 (see below).

Interlaboratory Study 89-2 (This Report)

This was the fourth (and final) interlaboratory study to validate the performance of the Method for Resin and Fatty Acids, Draft 3 (4). A set of nine samples was distributed to the participants in March 1989. The sample sets consisted of a High Spike, Low Spike, and Blank in Reagent Water, a High Spike, Low Spike, and Blank of Pulp and Paper Mill Effluent #1, a High Spike, Low Spike, and Blank of Pulp and Paper Mill Effluent #2.

The results indicated that the laboratories were able to recover the spiked Resin and Fatty Acids from the reagent water samples. Results from the two different spiked pulp and paper mill effluents produced very variable results, with very few laboratories reporting results from Pulp and Paper Mill Effluent #2. The same sources of pulp and paper mill effluent were used for both Interlaboratory Study 88-3 and this study (89-2). After reviewing the mill processes, it was noted that Pulp and Paper Mill Effluent #2 was a biologically treated effluent. The absence of recovery of the spiked Resin and Fatty Acids from this effluent in both studies, suggests that the effluent was still biologically active and degraded the spiking material.

Separate reports have been prepared for each study.

2 SUMMARY AND CONCLUSIONS

Interlaboratory Study 89-2 was the fourth and final study to validate the performance of the Method for Resin and Fatty Acids, Draft 3 (4). It was initiated by the Quality Assurance Office, Laboratory Services Branch of the Ontario Ministry of the Environment, at the request of the Analytical Working Group (AWG) of the Pulp and Paper Sector under the MISA program.

Participants were provided with a set of nine samples, consisting of a Blank, Low and High Spike in Reagent Water, a Blank, Low and High Spike in Pulp and Paper Mill Effluent #1, and a Blank, Low and High Spike in Pulp and Paper Mill Effluent #2. Eleven laboratories were invited to participate in this study. Results were reported from ten participants.

The results from this study demonstrate that the Draft Method is suitable for the analysis of samples of reagent water spiked with Resin and Fatty Acids. Consistent results may be obtained between laboratories, as long as the calibration of Dehydroabietic Acid is carried out in a precise and accurate manner.

The results from the spiked treated effluent samples were very variable. Low recoveries of the spiking material were observed from the four spiked effluent samples. Review of the treatment processes at the two pulp and paper mills suggest that the effluents may still have been biologically active and therefore degraded the spiking material. The same sources for the effluents were used for Interlaboratory Study 88-3 (7) and this study (Interlaboratory Study 89-2), with similar results observed for both sets of spiked effluent samples. Future work should attempt to establish the ability to recover Resin and Fatty Acids from different treated and untreated pulp and paper mill effluents. Future studies should also attempt to develop a suitable preservative to be used when collecting samples for Resin and Fatty Acid analysis.

3 INTRODUCTION

In June 1988, Interlaboratory Study 88-2A was conducted to validate the interlaboratory performance of the Method for Resin and Fatty Acids, Draft 3 (4), by the Quality Assurance Office, Laboratory Services Branch of the Ontario Ministry of the Environment. The results from this study have been reported elsewhere (5). A follow-up study was required to assess whether the different solvents used in the ampoule preparation affected the variability of the results. This second study was conducted in August 1988 and reported as Interlaboratory Study 88-2B (6). The results indicated that the solvent used to prepare the ampoules did not affect the variability of the results.

The Analytical Working Group (AWG) of the MISA Pulp and Paper Sector agreed to proceed with the next stage of the method validation. Interlaboratory Study 88-3 was designed to assess interlaboratory variability in the analysis of spiked reagent water and spiked pulp and paper mill effluents. Two treated pulp and paper mill effluents were provided for spiking with known amounts of Resin and Fatty Acids. A third untreated pulp and paper mill effluent was provided as an extra sample for between laboratory comparison. It was not spiked with the Resin and Fatty Acid standard solution used for the reagent water and two treated effluent samples. The results from Interlaboratory Study 88-3 (7) demonstrated that the Draft Method was suitable for the analysis of spiked reagent water samples and performed consistently between laboratories when sufficient Resin and Fatty Acids were present in the sample. The results from the spiked treated effluent samples were very variable and the AWG requested that the study be repeated.

Interlaboratory Study 89-2 was initiated as the repeat of Interlaboratory Study 88-3. The untreated effluent sample used in Study 88-3 was not included in Study 89-2. A new Resin and Fatty Acid Stock Solution was prepared for this study.

Eleven laboratories were invited to participate in this study: three government laboratories (provincial and federal), three industrial laboratories, and five commercial laboratories. All participants were required to agree to use the Draft Method for the analysis of the samples. If possible, the participants were asked to perform the analysis in duplicate. Not all of the participants were able to report final results for all of the samples (see Section 4.4). A list of invited participants is included in Appendix 2.

Details of sample preparation and distribution are given in Sections 4.1 and 4.2. Analytical methodology and data handling are presented in Sections 4.3 and 4.4. Final results are presented and discussed in Section 5.0. Each participant was assigned a unique identification code to maintain confidentiality.

4 PROCEDURE

4.1 Preparation of Samples

A 500 ppm Resin and Fatty Acid (RFA) solution, consisting of the parameters listed in Table 1, was prepared in methanol. The RFA solution was subsequently chilled to -20 degrees Celcius

prior to transferring to 5 mL amber ampoules. The solution was transferred via an automatic ampoule sealer and sealed with a flame. An Intermediate Spiking Solution was prepared in methanol by diluting an appropriate amount of the 500 ppm RFA Solution. Values for the final concentration were corrected for purity, based on the chemical supplier's information.

Deionized, distilled water was used as the matrix for the reagent water spikes. Care was taken that no plastic materials came into contact with the water when transferring to one litre amber sample bottles. To each bottle, 800 mL of water was added by weight ($800 \text{ g} \pm 2 \text{ g}$). Unspiked bottles were labelled "1A". The appropriate amount of RFA Intermediate Solution was dispensed into the bottles for the low and high spikes. The low spike was labelled "1B" and the high spike was labelled "1C".

Twenty litres of treated effluent was received from each of two different pulp and paper mills. The effluents were received in 2.5 litre bottles. All the effluent from one mill was poured into a clean, 50 litre, stainless steel tank, which had a spout. A stainless steel stirrer was used for one hour to homogenize the effluent. To each one litre amber sample bottle, 800 mL of effluent was transferred by weight ($800 \text{ g} \pm 2 \text{ g}$). The procedure was repeated for the second effluent. The appropriate amount of RFA Intermediate Solution was dispensed into the bottles for the low and high spikes. For Effluent #1 the samples were labelled as follows: unspiked - "2A", low spike - "2B", and high spike - "2C". For Effluent #2 the samples were labelled as follows: unspiked - "3A", low spike - "3B", and high spike - "3C".

TABLE 1 - Parameter List

Oleic Acid
Sandaracopimaric Acid
Isopimaric Acid
Palustric Acid
Levopimaric Acid
Dehydroabietic Acid
Abietic Acid
Neoabietic Acid
9,10-Dichlorostearic Acid
Chlorodehydroabietic Acid
Dichlorodehydroabietic Acid

4.2 Sample Distribution

The invited participants received a letter of notification in February, 1989. A copy is included in Appendix 2. All laboratories verified their participation by telephone. A list of invited participants is included in Appendix 2.

A set of nine samples, one each of 1A, 1B, 1C, 2A, 2B, 2C, 3A, 3B, and 3C, were packaged in boxes and shipped via Purolator courier on March 20, 1989. Each package included a cover letter and instruction sheet. Copies are included in Appendix 2.

4.3 Analytical Methodology

All participating laboratories were required to use the Draft Method for Resin and Fatty Acids (4). The analytical principles of this method are described in Schedule 2 of the Draft Pulp and Paper Regulation (2). All participants were asked to provide details of any modifications they may have made to the method. Each participant was asked to analyze each sample in duplicate, if possible.

4.4 Data Handling

Results were submitted to the QA Office of Laboratory Services Branch, MOE in written form by mail. All data were manually entered into an electronic spreadsheet. Blank spaces were left when a laboratory did not report results for a parameter that was present in the spiking material (i.e. the participant did not have the appropriate calibration standard). A value of "0" was used for the purpose of numerical evaluation for values reported as "Not Detected" by the laboratories.

One commercial laboratory did not report any results and did not provide any explanations as to why they were unable to submit results. Final percent participation was 91%.

Between-laboratory variability was determined by calculating the mean and standard deviation of the results reported. All results are presented in Appendix 1, Table 1.

Results were converted to percent recovery based on the design value of the spiking material. These values are presented in Appendix 1, Table 2.

Included in Appendix 1 are bar graphs of the recovery for each parameter from each participating laboratory (Figures 1-6). Each graph represents a different sample and the parameters are arranged left to right in order of gas chromatographic elution, using a DB-1 fused silica capillary column, as specified in the Draft Method (4). Outliers were not deleted from the data set when preparing the graphs. All results are presented as percent recovery relative to the design value. When a laboratory submitted duplicate results for a sample, the mean of the duplicates was used for these graphs.

In Interlaboratory Study 88-2A (5) it was observed that interlaboratory bias is introduced due to the differences in calibration of Dehydroabietic Acid (DHA). DHA is used as the reference point when calibrating the instrument, and all other analytical results are calculated using the relative response factor of this parameter (4). The percent recovery data from Samples 1B and 1C (Low and High Spikes in Reagent Water) were normalized relative to the results for DHA reported by each participant, to remove the effect of laboratory differences in calibration. The normalized results are presented in bar graphs for each parameter in Appendix 1 (Figures 7-15). This was not done for the spiked effluent samples (2B, 2C, 3B, and 3C) as there was insufficient data to do so.

In Interlaboratory Study 88-2A (5) one of the participants reported results using GC/MS instead of GC/FID, as specified in the Draft Method (4). This participant's data was excluded from the interpretation of the results. In this study, two participants provided two data sets, one using GC/FID as specified in the Draft Method, and one using GC/MS. The results using GC/MS are given in Table 3, Appendix 3. These results are provided for information purposes only, as they are not part of the validation of the Draft Method.

One of the industry laboratories also provided an additional data set. This participant uses a clean-up step, prior to instrumental analysis. These results are presented in Table 4, Appendix 3. These results are provided for information purposes only, as they are not part of the validation of the Draft Method.

5 RESULTS AND DISCUSSION

The raw results (Table 1, Appendix 1) were converted to percent recovery of the design values (Table 2, Appendix 1) to assess the performance of each participant. Preliminary evaluation of the results for the spiked Reagent Water samples (1B and 1C), suggest parameter or laboratory biases, similar to those observed for the percent recovery results in Interlaboratory Study 88-2A (5). To remove laboratory bias, the results from Samples 1B and 1C (High and Low Spikes, Reagent Water) were normalized relative to the value for Dehydroabietic Acid (DHA) reported by each participant.

The normalized results (Figures 7-15, Appendix 1) demonstrate consistent performance between laboratories for many of the parameters. Some parameters such as Neoabietic Acid (Figure 12) and 9,10-Dichlorostearic Acid (Figure 14) display variable performance. This may be due to the difficulty of obtaining pure, stable materials to prepare the spiking solutions, as has been observed previously (3, 5, 7). The normalized results from the low spike (1B) do not show any significant difference in pattern compared to the high spike (1C).

The results from the spiked effluent samples (2B, 2C, 3B, and 3C) displayed considerable variability.

Both of the treated effluents provided for the study were from pulp and paper mills that have biological treatment processes. This is the treatment process recommended for removal of Resin and Fatty Acid prior to discharging the effluent (2). The low recovery of the spiked material from Samples 2B, 2C, 3B, and 3C may be due to the effluents still being active and degrading the spiking material.

The results from this study demonstrate that the Draft Method is suitable for the analysis of samples of reagent water spiked with Resin and Fatty Acids. Differences between laboratories may be attributed to the difference in calibration of DHA. The accuracy of the results for other Resin and Fatty Acids is directly dependent on the precise and accurate calibration of DHA.

The variability of results from the spiked pulp and paper mill effluents, both in this study and from Study 88-3 (7), suggest that the matrix affects the recovery of Resin and Fatty Acids. In Study 88-3 (7), consistent results were observed from all but one of the participants, for the *untreated* pulp and paper mill effluent. The two *treated* effluents appeared to degrade the spiking material, making it difficult to assess interlaboratory performance of the method. Future studies should attempt to establish the ability to recover Resin and Fatty Acids from different treated and untreated pulp and paper mill effluents. Future work should also attempt to develop a suitable preservative to be used when collecting samples for Resin and Fatty Acid analysis.

6 REFERENCES

1. Ontario Regulation 695/88 as amended to Ontario Regulation 533/89 under the Environmental Protection Act; Effluent Monitoring - General.
2. The Development Document for the Draft Effluent Monitoring Regulation for the Pulp and Paper Sector, March 1989; ISBN 0-7729-4764-3.
3. Minutes from the OFIA/MOE/EC Analytical Working Group, 1987 and 1988, inclusive. (Copies of all minutes were reported to the Joint Technical Committee for the Pulp and Paper Sector.)
4. Method for Resin and Fatty Acids; OFIA/MOE/EC Analytical Working Group; Draft, May 5, 1988.
5. Interlaboratory Study 88-2A; Validation of a Method for Resin and Fatty Acids; Ampoules for Spiking Reagent Water and Direct Methylation; February 1990, ISBN 0-7729-6749-0.
6. Interlaboratory Study 88-2B; Validation of a Method for Resin and Fatty Acids; Ampouled Standards in Two Different Solvents for Direct Methylation and Instrumental Injection; February 1990, ISBN 0-7729-6750-4.
7. Interlaboratory Study 88-3; Validation of a Method for Resin and Fatty Acids; Spiked Samples Prepared in Reagent Water and Pulp and Paper Mill Effluents; February 1990, ISBN 0-7729-6751-2

7 APPENDIX 1 - FULL DATA SET

Table 1	Results for Interlaboratory Study 89-2
Table 2	Results for Interlaboratory Study 89-2, Expressed as Percent Recovery of the Design Value
Figure 1	Sample 1B - Low Spike, Reagent Water
Figure 2	Sample 1C - High Spike, Reagent Water
Figure 3	Sample 2B - Low Spike, Effluent #1
Figure 4	Sample 2C - High Spike, Effluent #1
Figure 5	Sample 3B - Low Spike, Effluent #2
Figure 6	Sample 3C - High Spike, Effluent #2
Figure 7	Oleic Acid Normalized to DHA (Samples 1B and 1C)
Figure 8	Sandaracopimaric Acid Normalized to DHA (Samples 1B and 1C)
Figure 9	Isopimaric Acid Normalized to DHA (Samples 1B and 1C)
Figure 10	Palustric & Levopimaric Acids Normalized to DHA (Samples 1B and 1C)
Figure 11	Abietic Acid Normalized to DHA (Samples 1B and 1C)
Figure 12	Neoabietic Acid Normalized to DHA (Samples 1B and 1C)
Figure 13	Chlorodehydroabietic Acid Normalized to DHA (Samples 1B and 1C)
Figure 14	9,10-Dichlorostearic Acid Normalized to DHA (Samples 1B and 1C)
Figure 15	Dichlorodehydroabietic Acid Normalized to DHA (Samples 1B and 1C)

TABLE 1 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
RESULTS EXPRESSED IN PPB

SAMPLE#	PARAMETER	DISTRIBUTED: MARCH 21, 1989	LABORATORY NUMBER										MEAN	RANGE		STD DEV
			6001	6002	6004	6005	6006	6007	6008	6009	6010	6011		MIN	MAX	
			RES	RES1	RES2	RES1	RES2	RES	RES	RES	RES	RES1	RES2			
1A	OLEIC			115				9.46								
	SANDAROPICINARIC															
	ISOPINARIC															
	PALUSTRIC/LEUOPINARIC															
	DEHYDROABIETIC															
	ABIETIC															
	NEOBIETIC															
	CHLORODEHYDROABIETIC			41												
	9,10-DICHLOROSTEARIC			23												
	DICHLORODEHYDROABIETIC															
	*PALMITIC															
	o-METHYLPODOCARPIC ACID (Z)		98.4Z	127.0Z				55.0Z	90.4Z	91.0Z	87.7Z					
	TRICOSANOIC ACID (Z)						102Z									
1B	OLEIC		50	31.3	345	260	18.4	16.4	41	20.3	30.1	51	41.7	58	58	103.0
	SANDAROPICINARIC		44.7	72.2	400	360	84.2	80.6	64	66.1	71.8	63	62.3	94	94	122.3
	ISOPINARIC		54.5	94.4	600	500	115	106	89	80.8	96.6	93	65.6	131	131	170.0
	PALUSTRIC/LEUOPINARIC		94.6	58.5	270	270	79.5	76.7	90	0	63.6	55.1	93	67.1	116	113
	DEHYDROABIETIC		50	106	650	550	137	131	115	156	109	102	93	86.4	138	138
	ABIETIC		47.2	60.9			79.9	76.6	77	30	58.9	59	80	52.2	106	103
	NEOBIETIC		50	17.5	24	38	58.5	55.3	58	0	38.3	0	55	34	79	77
	CHLORODEHYDROABIETIC		47.3	44.4	105	95	33.2	31.1	65	54	45.5	0	63	44.3	75	74
	9,10-DICHLOROSTEARIC		48	29.7	95	80	50	50	27	73	29	47	64	64	64	64
	DICHLORODEHYDROABIETIC		50	38	230	200	75.9	70.5	80	62	32.1	75.5	81	48.6	84	81
	*PALMITIC															
	*LINOLEIC															
	*LINOLENIC															
	o-METHYLPODOCARPIC ACID (Z)		99.4Z	118.0Z	104.0Z					48.0Z	89.0Z	90.0Z	85.0Z			
	TRICOSANOIC ACID (Z)									102Z						
	MEAN RESULTS WITHIN SCAN		55.3	302.1	261.4	73.2	69.4	71.0	56.9	54.4	55.3	71.9	55.8	94.5	94.5	93.3

* - ADDITIONAL PARAMETERS REPORTED BY SOME OF THE PARTICIPANTS
o-METHYLPODOCARPIC ACID/TRICOSANOIC ACID - SUBROGATE STANDARDS
REAGENT WATER: 1A - UNSPIRED; 1B - LOW SPIRE; 1C - HIGH SPIRE

RES1/RES2 - DUPLICATE RESULTS REPORTED BY SOME OF THE PARTICIPANTS

TABLE 1 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
RESULTS EXPRESSED IN ppb

DISTRIBUTED: MARCH 21, 1989																			
SAMPLE#	PARAMETER	DESIGN (ppb)	LABORATORY NUMBER										MEAN	RANGE MIN	RANGE MAX	STD DEV			
			6001 RES	6002 RES1	6002 RES2	6004 RES1	6004 RES2	6005 RES	6006 RES	6007 RES	6008 RES	6009 RES					6010 RES	6011 RES1	6011 RES2
2A	OLEIC	-				125													
	SANDARACOPINARIC	-																	
	ISOPINARIC	-																	
	PALUSTRIC/LEVOPIINARIC	-																	
	DEHYDROABIETIC	-				24													
	ABIETIC	-																	
	NEOABIETIC	-																	
	CHLORODEHYDROABIETIC	-				24													
	9,10-DICHLOROSTEARIC	-				24													
	DICHLORODEHYDROABIETIC	-																	
	MPALMITIC	-																	
	KLINOLENIC	-																	
	o-METHYLPODOCARPIC ACID (Z)			97.0Z	120.0Z														
	TRICOSANOIC ACID (Z)																		
2B	OLEIC	50	0	110	140	26.6	12.7	0	0	0	0	0	0	0	0	22.3	0.0	140.0	46.7
	SANDARACOPINARIC	44.7	0	33	37	0	0	0	0	0	21.5	38	9.9	25	25	14.6	0.0	38.0	15.7
	ISOPINARIC	54.5	0	0	0	0	0	0	0	0	27.7	53	9.5	30	29	11.5	0.0	53.0	17.5
	PALUSTRIC/LEVOPIINARIC	94.6	0	0	15	0	0	0	0	0	15.8	40	12.1	38	38	12.2	0.0	40.0	16.2
	DEHYDROABIETIC	50	0	35	40	0	0	1	0	0	68.6	81	39.9	66	64	30.4	0.0	81.0	31.8
	ABIETIC	47.2	6.94			0	0	0	0	0	0	57	16.9	36	35	13.8	0.0	57.0	20.0
	NEOABIETIC	50	0	0	0	0	0	0	0	0	0	26	9.7	33	33	7.8	0.0	33.0	13.4
	CHLORODEHYDROABIETIC	47.3	0	58	75	0	0	1	0	0	0	58	21.5	48	46	23.7	0.0	75.0	28.8
	9,10-DICHLOROSTEARIC	48	0	21	27	0	0	0	0	0	0	0	0	0	0	4.4	0.0	27.0	9.8
	DICHLORODEHYDROABIETIC	50	15.6	135	155	21	13.4	1	0	0	52.2	64	13.4	58	57	45.0	0.0	155.0	50.3
	MPALMITIC																		
	o-METHYLPODOCARPIC ACID (Z)			96.4Z	120.0Z	140.0Z													
	TRICOSANOIC ACID (Z)																		
	MEAN RESULTS WITHIN SCAN			2.3	43.6	54.3	4.8	2.6	0.3	0.0	0.0	20.6	41.7	14.8	33.4	32.7			

* - ADDITIONAL PARAMETERS REPORTED BY SOME OF THE PARTICIPANTS
o-METHYLPODOCARPIC ACID/TRICOSANOIC ACID - SURROGATE STANDARDS
PULP AND PAPER EFFLUENT #1: 2A - UNSPIKE; 2B - LOW SPIKE; 2C - HIGH SPIKE
RES1/RES2 - DUPLICATE RESULTS REPORTED BY SOME OF THE PARTICIPANTS

TABLE 1 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS RESULTS EXPRESSED IN PPB

[illegible]

DISCREPANCY - DUPLICATE RESULTS REPORTED BY SOME OF THE PARTICIPANTS

* - ADDITIONAL PARAMETERS REPORTED BY SOME OF THE PARTICIPANTS
o-METHYLPICOLIC ACID/TRICLOSANIC ACID - SUBSTRATE STANDARDS
HPLP AND PAPER EFFLUENT #1: 2B - UNSPLITTED; 2B - 100 SPIKE; 2C -

TABLE 1 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
RESULTS EXPRESSED IN PPB

DISTRIBUTED: MARCH 21, 1989		SAMPLE#		PARAMETER		DESIGN (ppb)		6001		6002		LABORATORY NUMBER		6005		6006		6007		6008		6009		6010		6011		MEAN		RANGE		STD DEV			
								RES		RES1		RES2		6004		RES		RES		RES		RES		RES		RES1		RES2		MIN		MAX			
3A	OLEIC	-	-	-	-	-	-	12.4	120			18.5	12.6												1.6	21	27								
	SANDARACOPIMARIC	-	-	-	-	-	-																	1.5	14	23	9								
	ISOPIMARIC	-	-	-	-	-	-	8.41																	6	10	18	10							
	PALUSTRIC/LEVOPIMARIC	-	-	-	-	-	-																												
	DEHYDROBIETIC	-	-	-	-	-	-	8.41	45															3.9	11	9	6								
	ABIETIC	-	-	-	-	-	-	8.41																											
	NEOBIEETIC	-	-	-	-	-	-																												
	CHLORODEHYDROBIETIC	-	-	-	-	-	-																												
	9,10-DICHLOROSTEARIC	-	-	-	-	-	-																												
	DICHLORODEHYDROBIETIC	-	-	-	-	-	-	-																											
3B	*PALMITIC	-	-	-	-	-	-																												
	*STEARIC	-	-	-	-	-	-																												
	*LINOLEIC	-	-	-	-	-	-																												
	*LINOLENIC	-	-	-	-	-	-																												
	*PIMARIC	-	-	-	-	-	-																												
	o-METHYLPODOCARPIC ACID (Z)							97.4Z	107.0Z																										
	TRICOSANOIC ACID (Z)																																		
	OLEIC	50	10.7	175	13.1	29.5	2	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	16.9	36	35								
	SANDARACOPIMARIC	44.7	0	10	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	18	8	0							
	ISOPIMARIC	54.5	7.07	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	33	2.5	6	6	6	4.0	0.0	33.0	9.4		

RES1/RES2 - DUPLICATE RESULTS REPORTED BY SOME OF THE PARTICIPANTS

* - ADDITIONAL PARAMETERS REPORTED BY SOME OF THE PARTICIPANTS
o-METHYLPODOCARPIC ACID/TRICOSANOIC ACID - SUBSTRATE STANDARDS
PULP AND PAPER EFFLUENT #2: 30 - UNSPIKED; 30 - LOW SPIKE; 30 - HIGH SPIKE

DISTRIBUTED: MARCH 21, 1989
SAMPLE# PARAMETER

TABLE 1 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
RESULTS EXPRESSED IN PPB

DESIGN SAMPLE#	PARAMETER	6001 RES	6002 RES1 RES2	LABORATORY NUMBER 6004 RES1 RES2	6005 RES	6006 RES	6007 RES	6008 RES	6009 RES	6010 RES	6011 RES1 RES2	MEAN	RANGE MIN MAX	STD DEV
3C	OLEIC	400	7.66	180	190	15.4	14.1	0	0	0	40	37.6	0.0 190.0	66.9
	SANDARACOPIMARIC	357.6	0	16	17	0	0	0	0	136	29	17.4	0.0 136.0	37.2
	ISOPIMARIC	436	12.7	95	100	0	0	0	0	253	17	16	0.0 253.0	73.2
	PHLOSTRIC/LEVOPIMARIC	756.8	0	24	26	0	0	0	0	101	12	13	0.0 101.0	27.9
	DEHYDROABETIC	400	7.66	91	68	0	0	0	0	377	22	20	0.0 377.0	103.7
	ABETIC	377.6	7.66	12	75	17.5	17.6	0	0	193	23	21	0.0 193.0	57.1
	NEOABETIC	400	0	120	135	0	0	0	0	112	0	0	0.0 112.0	34.7
	CHLORODEHYDROABETIC	378.4	0	32	31	0	0	0	0	238	15	27	0.0 238.0	74.9
	9,10-DICHLOROSTEARIC	364	0	265	270	69.6	71.4	0	0	16	7	13	0.0 32.0	12.5
	DICHLORODEHYDROABETIC	400	28	265	270	69.6	71.4	134	273	61.2	72	67	28.0 400.0	113.6
	*PALMITIC							50	30.3		19	17		
	*STEARIC										13	10		
	*LINOLEIC							11.6			16	15		
	*LINOLENIC										11	10		
	*PIMARIC										19	14		
	o-METHYLPODOCARPIC ACID (%)	96.9%	128.0%	127.0%				82.0%	88.4%	89.0%	78.0%			
	TRICOSANOIC ACID (%)							102%						
	MEAN RESULTS WITHIN SCAN	6.4	92.8	101.3	10.4	10.3	4.3	5.0	3.0	14.9	169.9	10.9	24.5	24.0

* - ADDITIONAL PARAMETERS REPORTED BY SOME OF THE PARTICIPANTS
o-METHYLPODOCARPIC ACID/TRICOSANOIC ACID - SUBSTRATE STANDARDS
PULP AND PAPER EFFLUENT 12: 3A - UNSPIRED; 3B - LOW SPIRE; 3C - HIGH SPIRE
RES1/RES2 - DUPLICATE RESULTS REPORTED BY SOME OF THE PARTICIPANTS

TABLE 2 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
RESULTS EXPRESSED AS PERCENT RECOVERY OF DESIGN VALUE

LABORATORY NUMBER

DISTRIBUTED: MARCH 21, 1989

SAMPLE#	PARAMETER	DESIGN (ppb)	6001		6002		6004		6005		6006		6007		6008		6009		6010		6011		MEAN	RANGE	
			%REC	%REC1	%REC2	%REC1	%REC2	%REC	%REC1	%REC2	%REC	%REC1	%REC2	%REC	%REC1	%REC2	%REC	%REC1	%REC2	%REC	%REC1	%REC2		MIN	MAX
1B	OLEIC	50	63%	690%	520%	37%	33%	24%	82%	41%	76%	102%	83%	116%	116%	123%	24%	605%							
	SANDAROCOPIMARIC	44.7	162%	895%	805%	188%	180%	174%	143%	148%	161%	141%	139%	210%	210%	231%	139%	850%							
	ISOPIMARIC	54.5	173%	1101%	917%	211%	194%	198%	163%	148%	177%	171%	120%	240%	240%	260%	120%	1003%							
	PALUSTRIC/LEVOPIMARIC	94.6	62%	285%	285%	84%	81%	95%	0%	67%	58%	98%	71%	123%	119%	94%	0%	285%							
	DEHYDROABIETIC	50	212%	1300%	1100%	274%	262%	230%	312%	218%	204%	186%	173%	276%	276%	328%	173%	1200%							
	ABIETIC	47.2	129%																						
	NEOABIETIC	50	35%	48%	76%	117%	111%	116%	0%	77%	0%	110%	68%	158%	154%	74%	0%	156%							
	CHLORODEHYDROABIETIC	47.3	94%	222%	201%	70%	66%	137%	114%	96%	0%	133%	94%	159%	156%	111%	0%	211%							
	9,10-DICHLOROSTEARIC	48	62%	198%	167%	104%	104%	56%	152%	60%	98%	98%	133%	133%	106%	56%	182%								
DICHLORODEHYDROABIETIC	50	76%	460%	400%	152%	141%	160%	124%	64%	151%	162%	97%	168%	162%	158%	64%	430%								
MEAN RECOVERY WITHIN SCAN			107%	578%	497%	141%	133%	135%	115%	104%	106%	137%	106%	181%	179%										
1C	OLEIC	400	39%	225%		42%	36%	21%	40%	25%	35%	44%	41%	82%	82%	51%	8%	225%							
	SANDAROCOPIMARIC	357.6	69%	364%		85%	88%	98%	82%	72%	68%	69%	69%	93%	97%	107%	68%	364%							
	ISOPIMARIC	436	72%	413%		92%	94%	109%	93%	75%	71%	73%	66%	102%	109%	117%	66%	413%							
	PALUSTRIC/LEVOPIMARIC	756.8	31%	159%		44%	45%	59%	24%	31%	27%	45%	34%	51%	56%	51%	24%	159%							
	DEHYDROABIETIC	400	97%	550%		131%	135%	124%	147%	108%	103%	93%	70%	130%	135%	156%	70%	550%							
	ABIETIC	377.6	57%			81%	82%	96%	51%	59%	53%	73%	55%	96%	97%	62%	0%	97%							
	NEOABIETIC	400	24%	103%		57%	58%	71%	21%	35%	4%	56%	32%	64%	67%	47%	4%	103%							
	CHLORODEHYDROABIETIC	378.4	41%	74%		33%	34%	73%	72%	47%	105%	56%	42%	67%	67%	61%	34%	105%							
	9,10-DICHLOROSTEARIC	384	32%	96%		61%	55%	35%	53%	29%	53%	53%	59%	59%	52%	29%	96%								
DICHLORODEHYDROABIETIC	400	36%	200%		58%	60%	72%	65%	42%	58%	61%	39%	66%	64%	70%	36%	200%								
MEAN RECOVERY WITHIN SCAN			50%	243%		68%	69%	76%	65%	52%	58%	62%	50%	74%	76%										

REAGENT WATER: 1A - UNSPIKED; 1B - LOW SPIKE; 1C - HIGH SPIKE
%REC1/%REC2 - DUPLICATE RESULTS REPORTED BY SOME OF THE PARTICIPANTS

TABLE 2 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
RESULTS EXPRESSED AS PERCENT RECOVERY OF DESIGN VALUE

SAMPLE#	PARAMETER	LABORATORY NUMBER														MEAN	RANGE	
		DESIGN 6001 (ppb)	6002 %REC	6002 %REC1	6002 %REC2	6004 %REC1	6004 %REC2	6005 %REC	6006 %REC	6007 %REC	6008 %REC	6009 %REC	6010 %REC	6011 %REC	6011 %REC2		MIN	MAX
2B	OLEIC	50	0%	220%	280%	53%	25%	0%	0%	0%	0%	0%	0%	0%	0%	29%	0%	250%
	SANDAROCOPIMARIC	44.7	0%	74%	83%	0%	0%	0%	0%	0%	0%	48%	85%	22%	56%	29%	0%	85%
	ISOPIMARIC	54.5	0%	0%	0%	0%	0%	0%	0%	0%	0%	51%	97%	17%	55%	22%	0%	97%
	PALUSTRIC/LEVOPIMARIC	94.6	0%	0%	16%	0%	0%	0%	0%	0%	0%	17%	42%	13%	40%	12%	0%	42%
	DEHYDROABIETIC	50	0%	70%	80%	0%	0%	2%	0%	0%	0%	137%	162%	80%	132%	59%	0%	162%
	ABIETIC	47.2	15%	0%	0%	0%	0%	0%	0%	0%	0%	0%	121%	36%	76%	74%	0%	121%
	NEOABIETIC	50	0%	0%	0%	0%	0%	0%	0%	0%	0%	0%	52%	19%	66%	25%	0%	66%
	CHLORODEHYDROABIETIC	47.3	0%	123%	159%	0%	0%	2%	0%	0%	0%	0%	123%	45%	101%	41%	0%	141%
	9,10-DICHLOROSTEARIC	48	0%	44%	56%	0%	0%	0%	0%	0%	0%	0%	0%	0%	0%	6%	0%	50%
	DICHLORODEHYDROABIETIC	50	31%	270%	310%	42%	27%	2%	0%	0%	0%	104%	128%	27%	116%	73%	0%	290%
MEAN RECOVERY WITHIN SCAN			5%	89%	109%	10%	5%	1%	0%	0%	0%	40%	81%	29%	64%	63%		
2C	OLEIC	400	0%	43%	43%	4%	5%	0%	0%	0%	0%	0%	14%	37%	2%	10%	0%	43%
	SANDAROCOPIMARIC	357.6	28%	266%	266%	21%	20%	43%	33%	4%	0%	59%	70%	54%	73%	65%	4%	266%
	ISOPIMARIC	436	30%	310%	310%	24%	23%	48%	36%	5%	61%	71%	41%	41%	82%	71%	5%	310%
	PALUSTRIC/LEVOPIMARIC	756.8	4%	42%	42%	0%	0%	15%	0%	0%	19%	36%	19%	25%	25%	16%	0%	42%
	DEHYDROABIETIC	400	23%	275%	275%	0%	0%	58%	32%	1%	75%	92%	67%	94%	97%	72%	0%	275%
	ABIETIC	377.6	19%	48%	48%	7%	6%	41%	16%	3%	46%	73%	0%	69%	72%	27%	0%	48%
	NEOABIETIC	400	12%	48%	48%	16%	15%	37%	10%	6%	7%	54%	25%	53%	54%	27%	6%	73%
	CHLORODEHYDROABIETIC	378.4	23%	79%	79%	12%	12%	52%	36%	14%	75%	58%	37%	63%	63%	45%	12%	79%
	9,10-DICHLOROSTEARIC	384	4%	16%	16%	9%	7%	13%	0%	2%	2%	29%	9%	9%	9%	10%	0%	29%
	DICHLORODEHYDROABIETIC	400	24%	213%	213%	58%	52%	47%	32%	25%	48%	57%	41%	56%	57%	60%	24%	213%
MEAN RECOVERY WITHIN SCAN			17%	143%	143%	15%	14%	35%	19%	6%	43%	55%	36%	52%	54%			

PULP AND PAPER EFFLUENT #1: 2A - UNSPIKED; 2B - LOW SPIKE; 2C - HIGH SPIKE
%REC1/%REC2 - DUPLICATE RESULTS REPORTED BY SOME OF THE PARTICIPANTS

TABLE 2 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
RESULTS EXPRESSED AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: MARCH 21, 1989

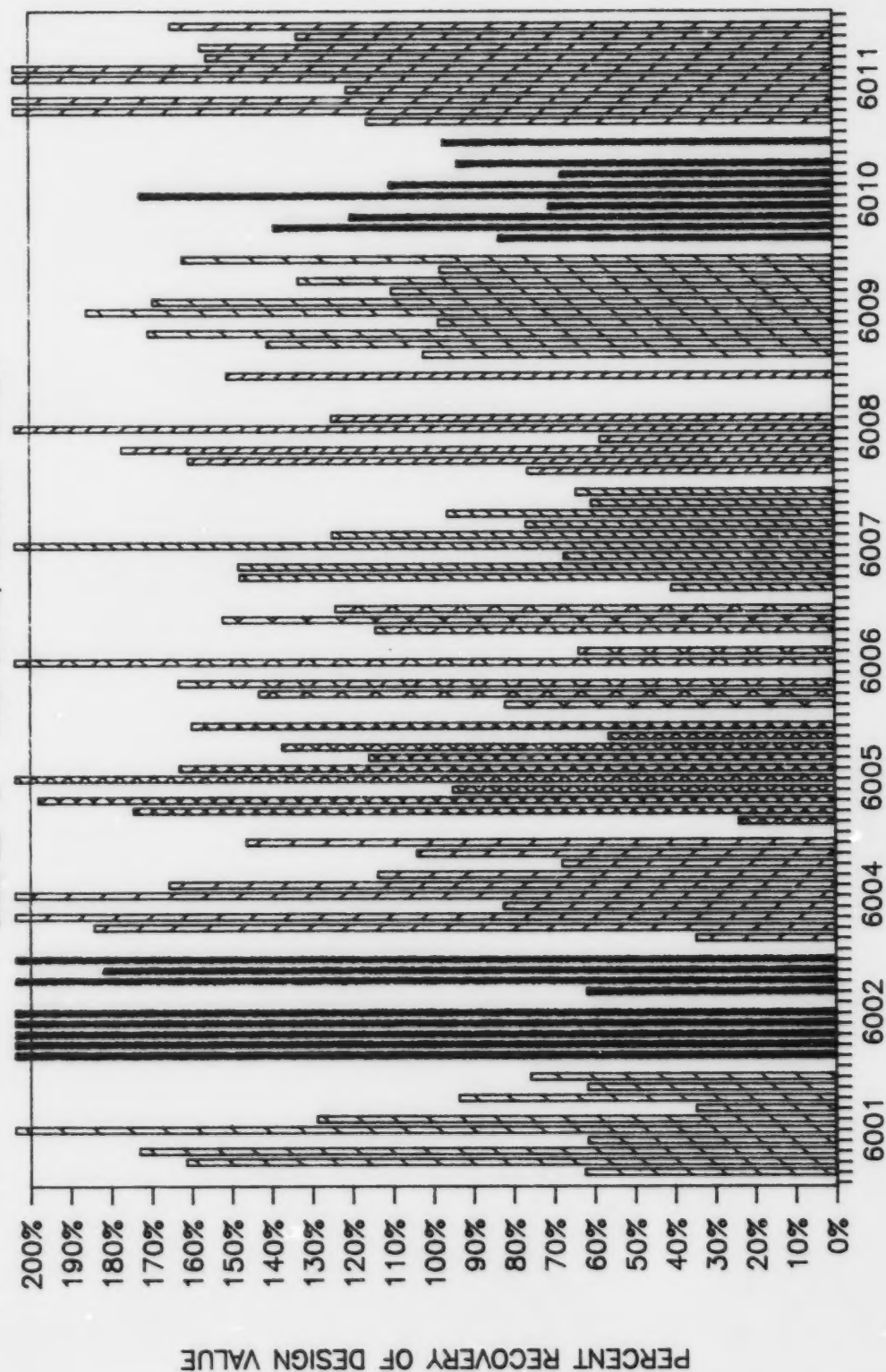
SAMPLE#	PARAMETER	LABORATORY NUMBER															
		DESIGN (ppb)	6001 %REC	6002 %REC1 %REC2	6004 %REC1 %REC2	6005 %REC %REC2	6006 %REC %REC2	6007 %REC %REC2	6008 %REC %REC2	6009 %REC %REC2	6010 %REC %REC2	6011 %REC1 %REC2	MEAN	RANGE MIN MAX			
3B	OLEIC	50	21%	350%	26%	59%	4%	0%	0%	0%	34%	72%	70%	52%	0%	350%	
	SANDARACOPIMARIC	44.7	0%	22%	0%	0%	0%	0%	0%	0%	29%	0%	0%	5%	0%	29%	
	ISOPIMARIC	54.5	13%	0%	0%	0%	0%	0%	0%	126%	0%	33%	15%	17%	0%	128%	
	PALUSTRIC/LEVOPIMARIC	94.6	0%	0%	0%	0%	0%	0%	0%	35%	3%	6%	6%	4%	0%	35%	
	DEHYDROABIETIC	50	14%	110%	0%	0%	0%	0%	0%	214%	0%	32%	18%	36%	0%	214%	
	ABIETIC	47.2	15%	0%	0%	0%	0%	0%	0%	133%	9%	30%	17%	18%	0%	133%	
	NEOABIETIC	50	0%	0%	0%	0%	0%	0%	0%	116%	0%	0%	0%	12%	0%	116%	
	CHLORODEHYDROABIETIC	47.3	0%	254%	0%	0%	0%	0%	0%	211%	0%	63%	13%	50%	0%	254%	
	9,10-DICHLOROSTEARIC	48	0%	98%	0%	0%	0%	0%	0%	0%	0%	38%	0%	15%	0%	98%	
	DICHLORODEHYDROABIETIC	50	14%	134%	0%	0%	0%	0%	15%	0%	270%	19%	34%	16%	48%	0%	270%
MEAN RECOVERY WITHIN SCAN			8%	108%	3%	6%	0%	2%	0%	114%	7%	31%	15%				
3C	OLEIC	400	2%	45%	48%	4%	4%	0%	0%	0%	1%	10%	9%	6%	0%	46%	
	SANDARACOPIMARIC	357.6	0%	4%	5%	0%	0%	0%	0%	38%	1%	8%	7%	5%	0%	38%	
	ISOPIMARIC	436	3%	22%	23%	0%	1%	0%	0%	58%	1%	4%	4%	9%	0%	58%	
	PALUSTRIC/LEVOPIMARIC	756.8	0%	3%	3%	0%	0%	0%	0%	13%	0%	2%	2%	2%	0%	13%	
	DEHYDROABIETIC	400	2%	23%	17%	0%	0%	0%	0%	94%	2%	6%	5%	12%	0%	94%	
	ABIETIC	377.6	2%	3%	19%	0%	0%	0%	0%	51%	2%	6%	6%	6%	0%	51%	
	NEOABIETIC	400	0%	32%	36%	0%	4%	0%	0%	28%	0%	2%	0%	4%	0%	28%	
	CHLORODEHYDROABIETIC	378.4	0%	8%	8%	0%	0%	0%	0%	63%	4%	4%	7%	11%	0%	63%	
	9,10-DICHLOROSTEARIC	384	0%	66%	68%	0%	0%	0%	0%	4%	0%	2%	3%	2%	0%	63%	
	DICHLORODEHYDROABIETIC	400	7%	23%	25%	17%	18%	10%	8%	34%	68%	15%	18%	17%	26%	0%	68%
MEAN RECOVERY WITHIN SCAN			2%	23%	25%	3%	3%	1%	1%	4%	42%	3%	6%				

PULP AND PAPER EFFLUENT #2: 3B - UNSEPTIC; 3C - LOW SPINE; 3E - HIGH SPINE
%REC1/%REC2 - DUPLICATE RESULTS REPORTED BY ONE OF THE PARTICIPANTS



FIGURE 1: INTERLABORATORY STUDY 89-2

SAMPLE 1B - LOW SPIKE, REAGENT WATER



FOR PARAMETER LIST, SEE FIGURE 2



FIGURE 2: INTERLABORATORY STUDY 89-2

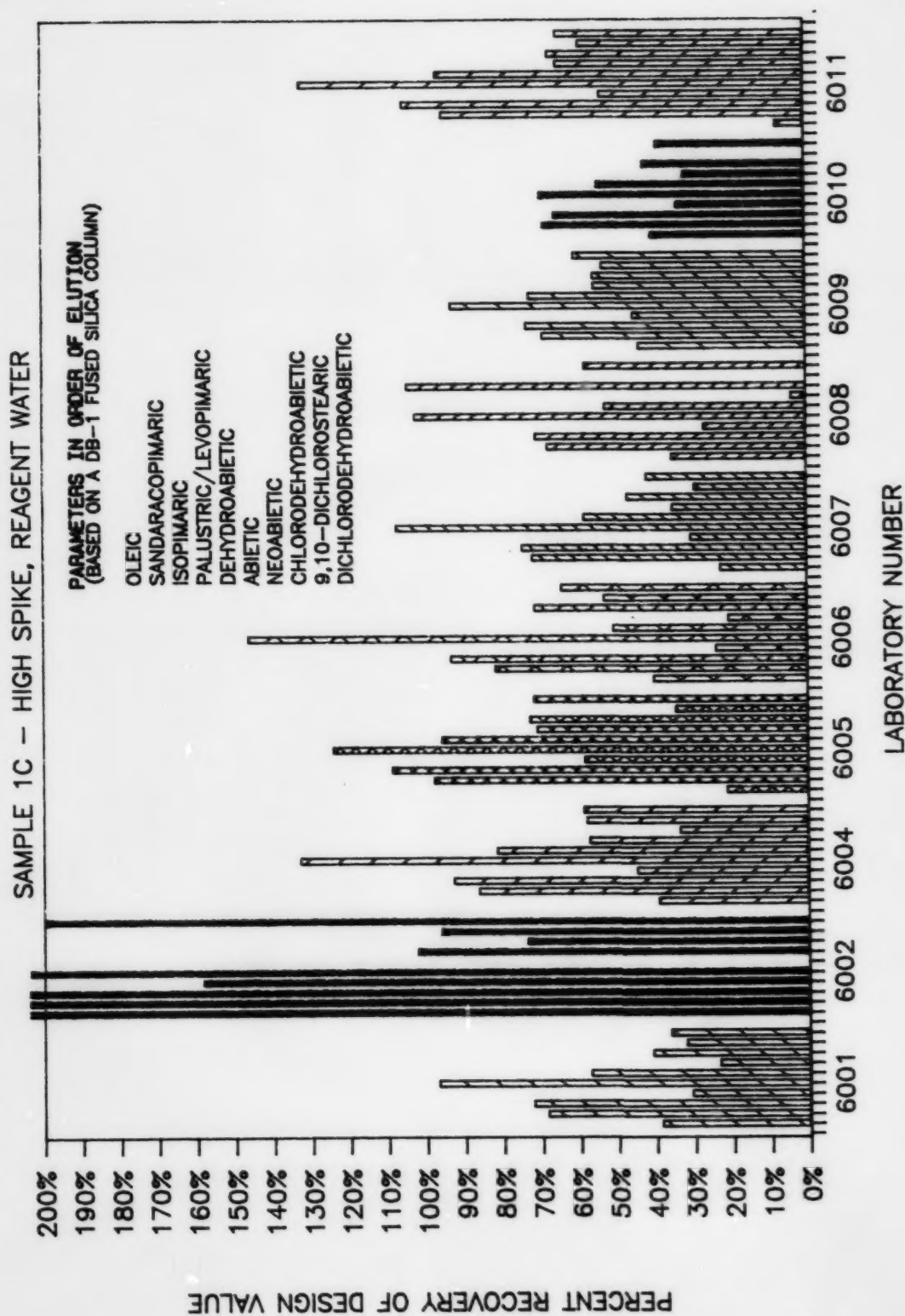


FIGURE 3: INTERLABORATORY STUDY 89-2

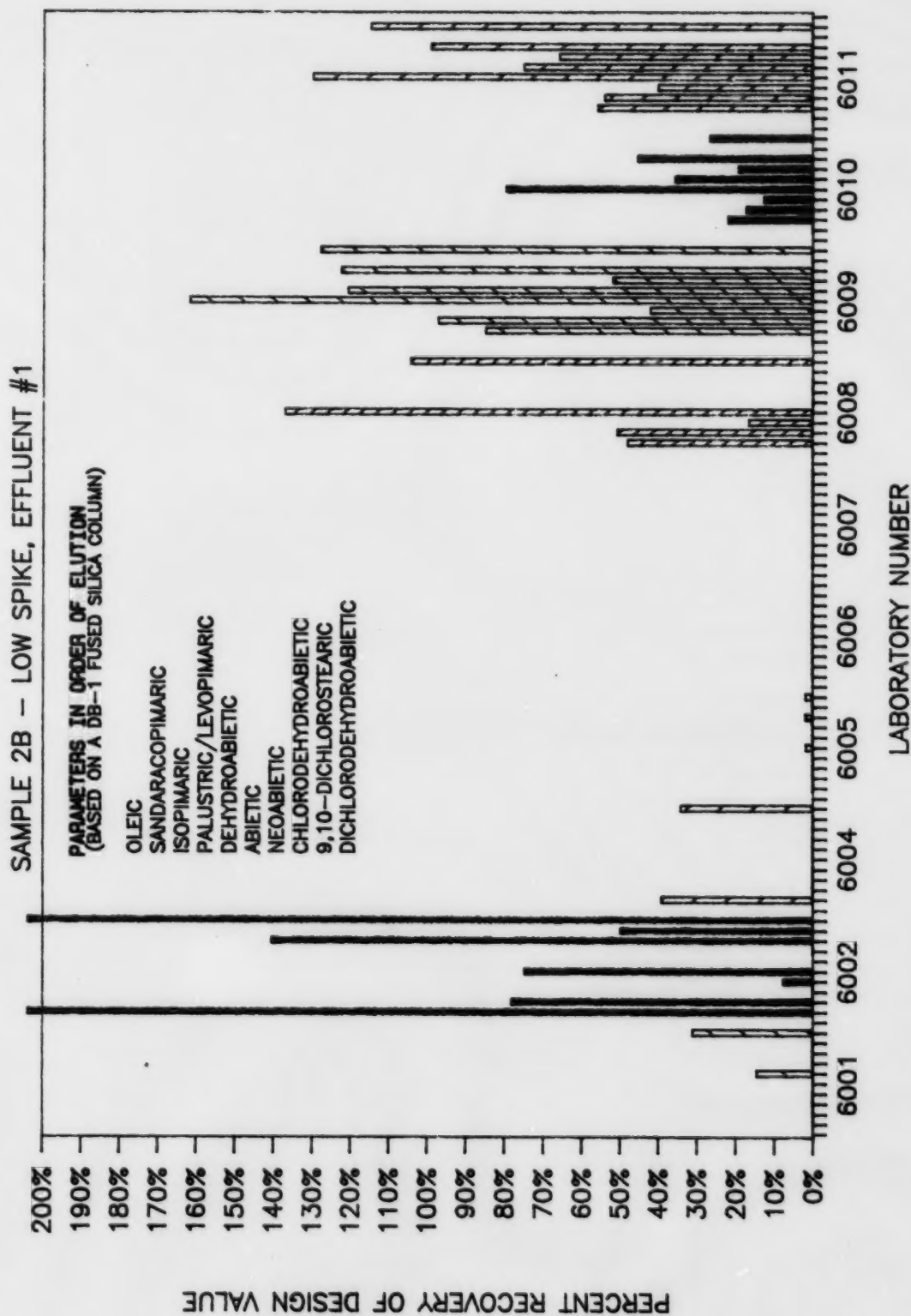




FIGURE 4: INTERLABORATORY STUDY 89-2

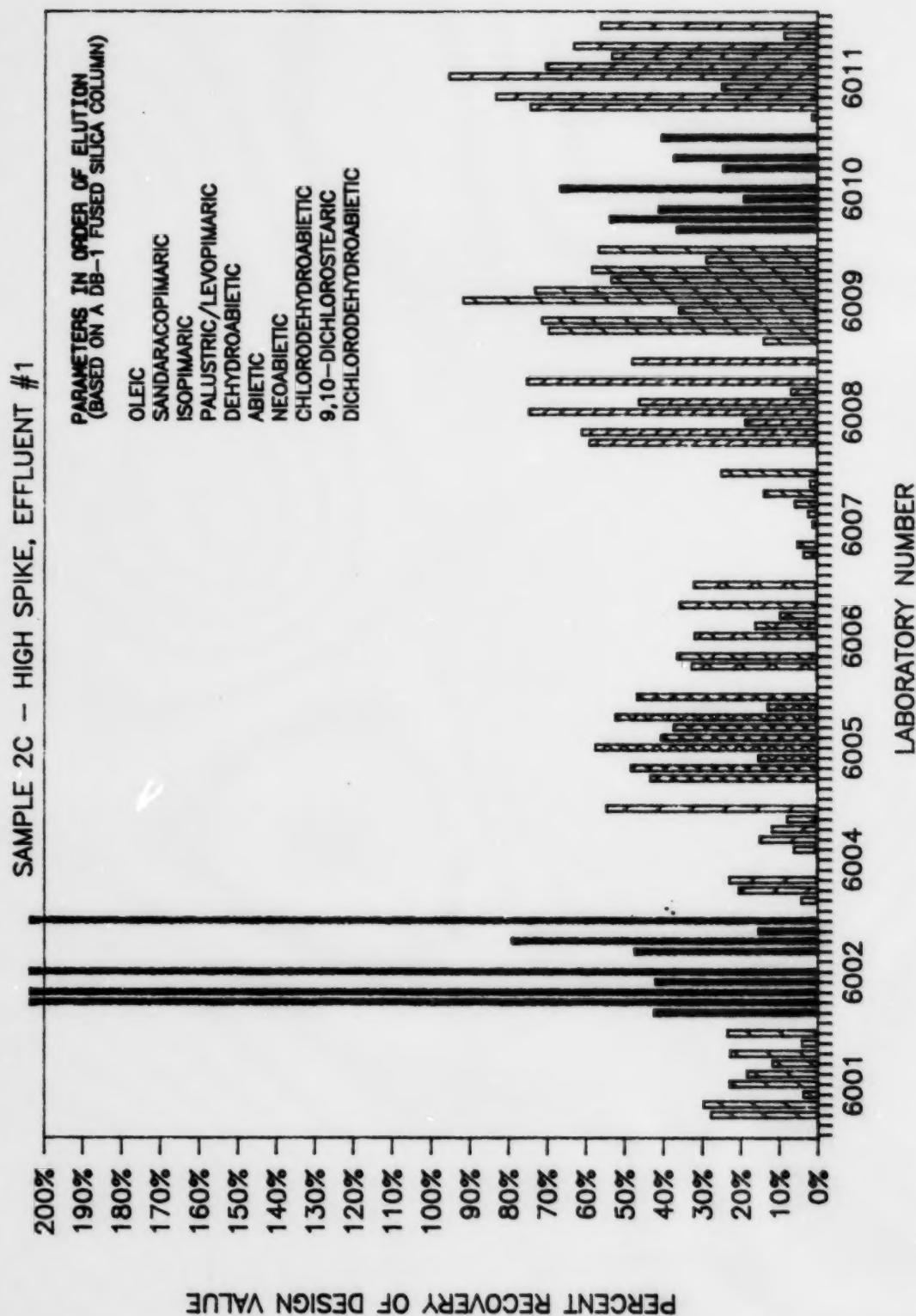




FIGURE 5: INTERLABORATORY STUDY 89-2

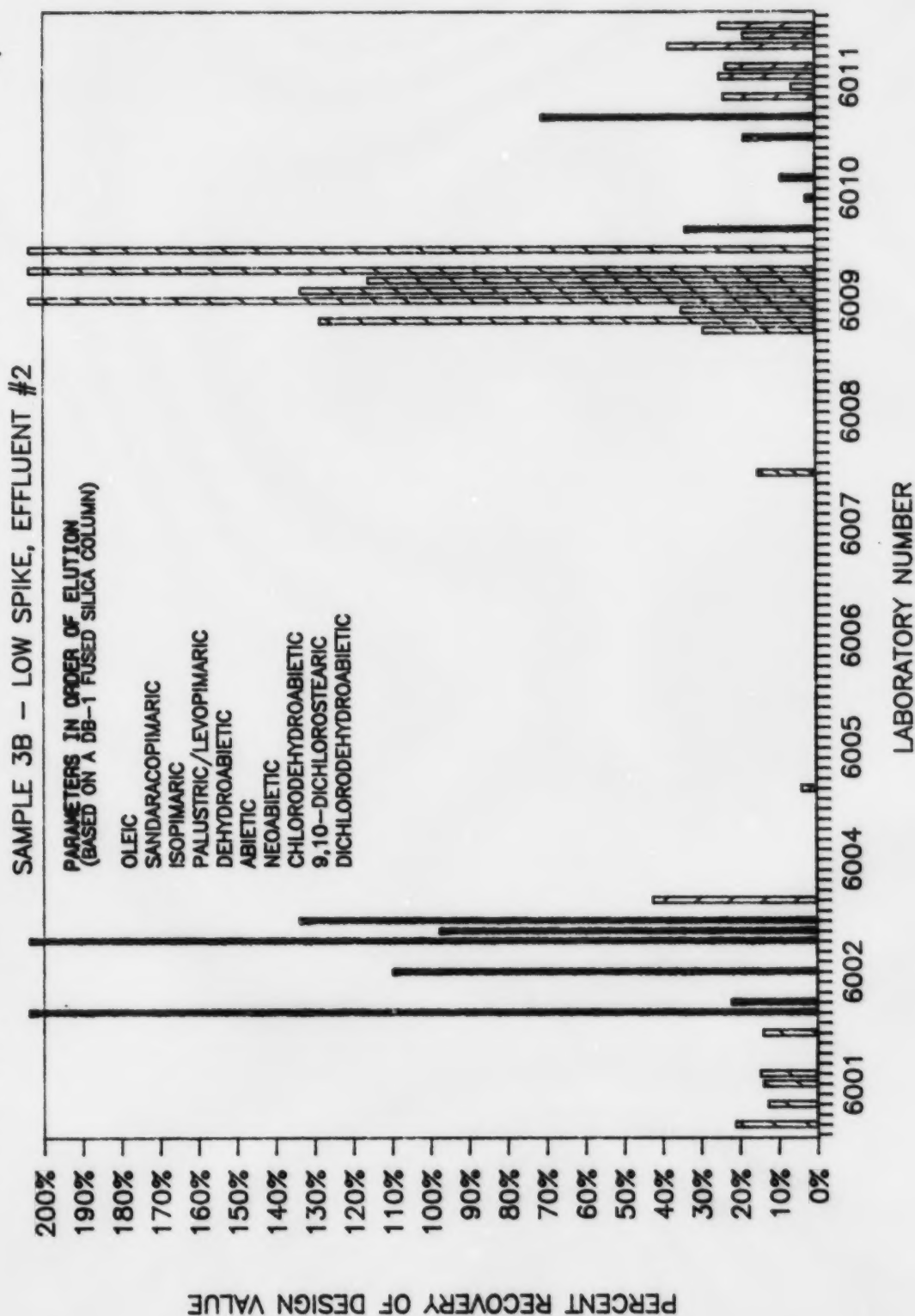




FIGURE 6: INTERLABORATORY STUDY 89-2

SAMPLE 3C - HIGH SPIKE, EFFLUENT #2

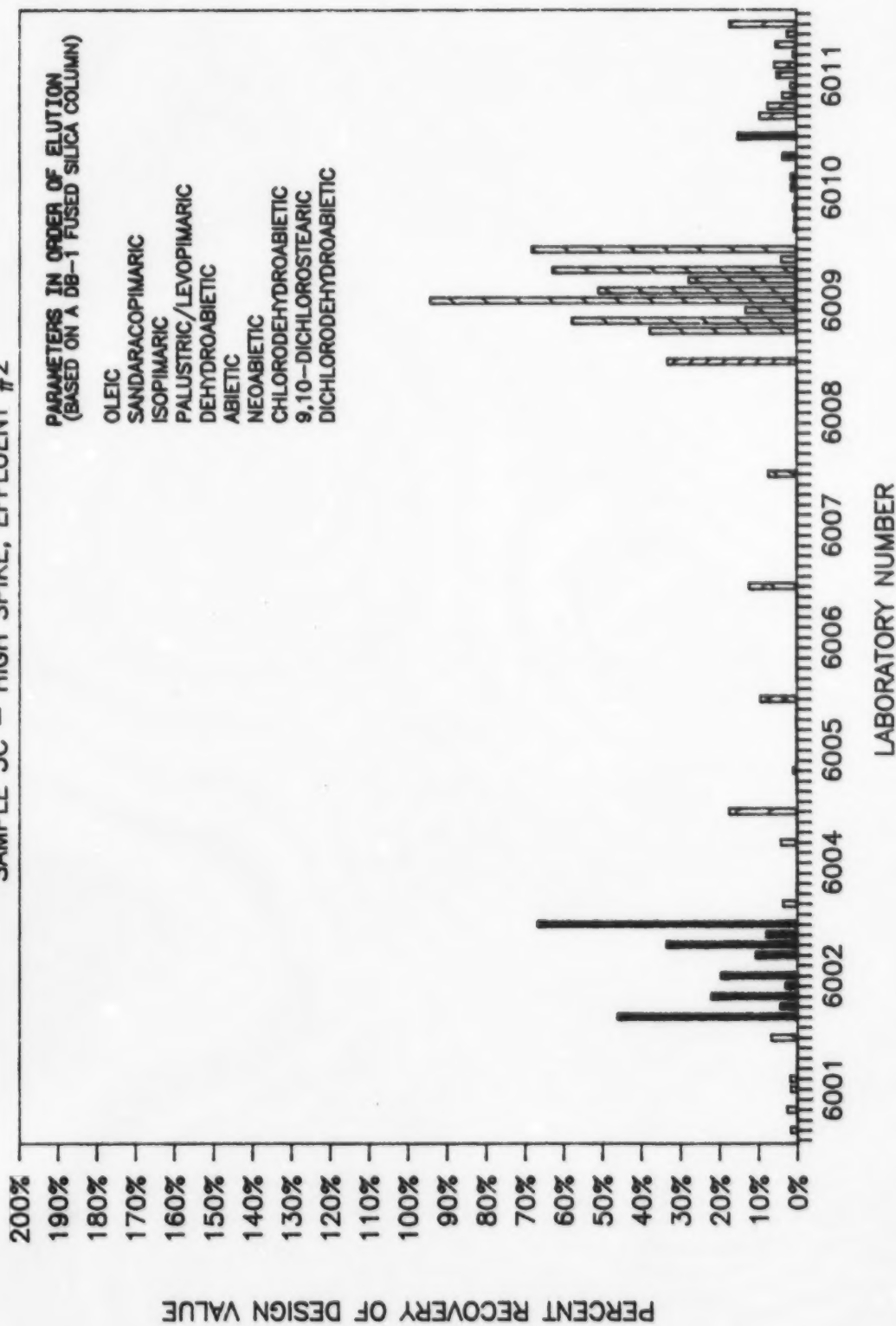


FIGURE 7: OLEIC ACID

RESULTS NORMALIZED TO DEHYDROABETIC

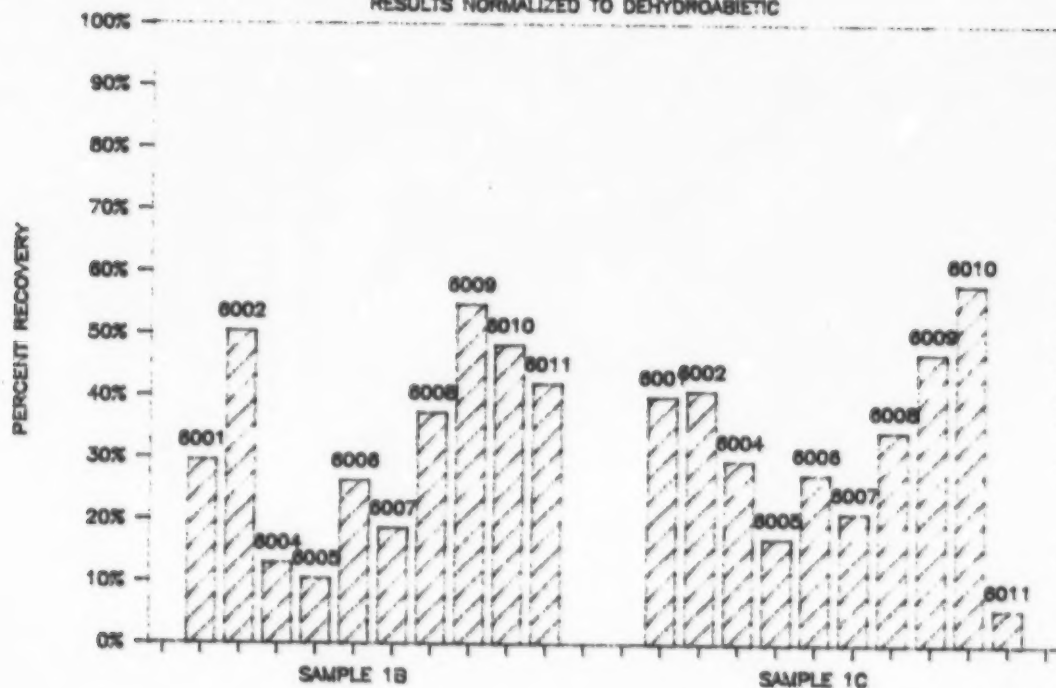


FIGURE 8: SANDARACOPIMARIC ACID

RESULTS NORMALIZED TO DEHYDROABETIC

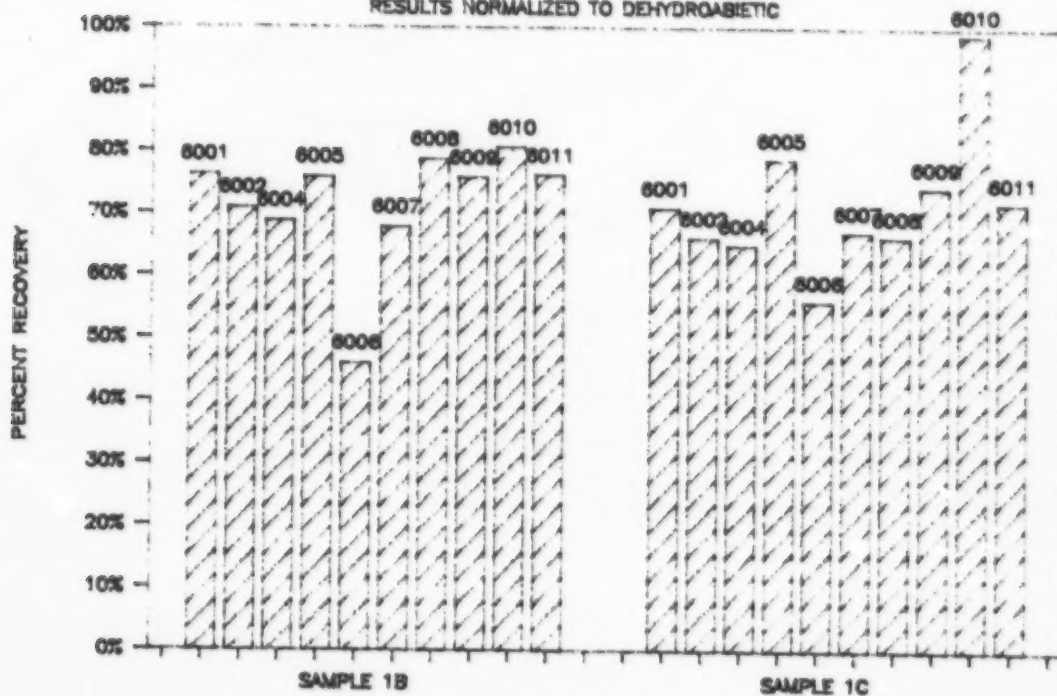


FIGURE 9: ISOPIMARIC ACID

RESULTS NORMALIZED TO DEHYDROABIETIC

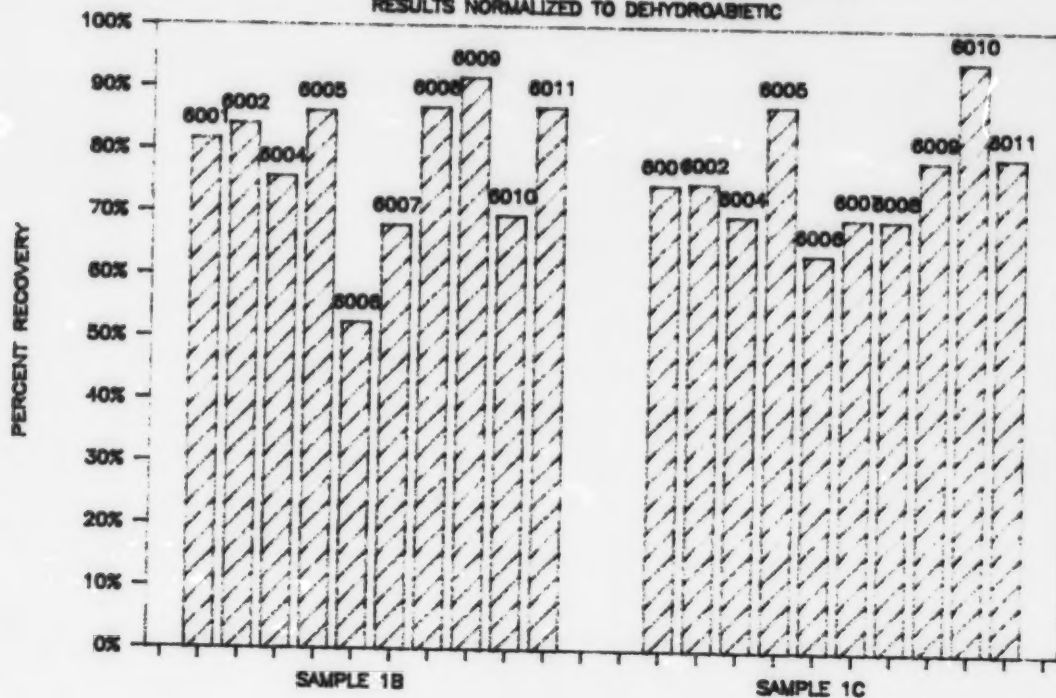


FIGURE 10: PALUSTRIC & LEVOPIMARIC ACID

RESULTS NORMALIZED TO DEHYDROABIETIC

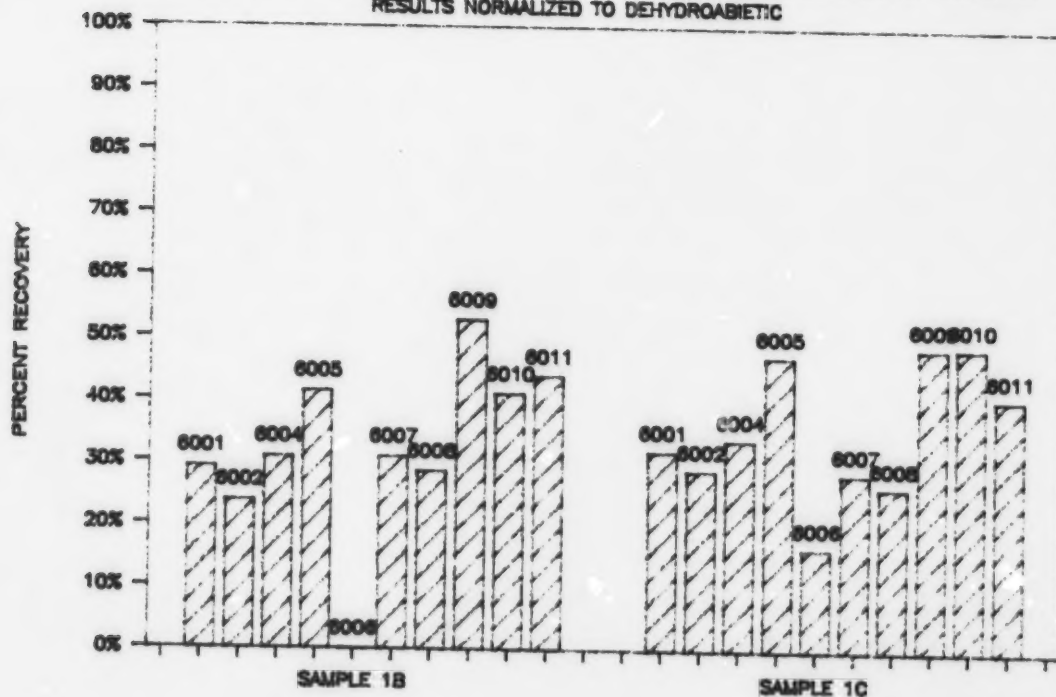


FIGURE 11: ABIETIC ACID

RESULTS NORMALIZED TO DEHYDROABIETIC

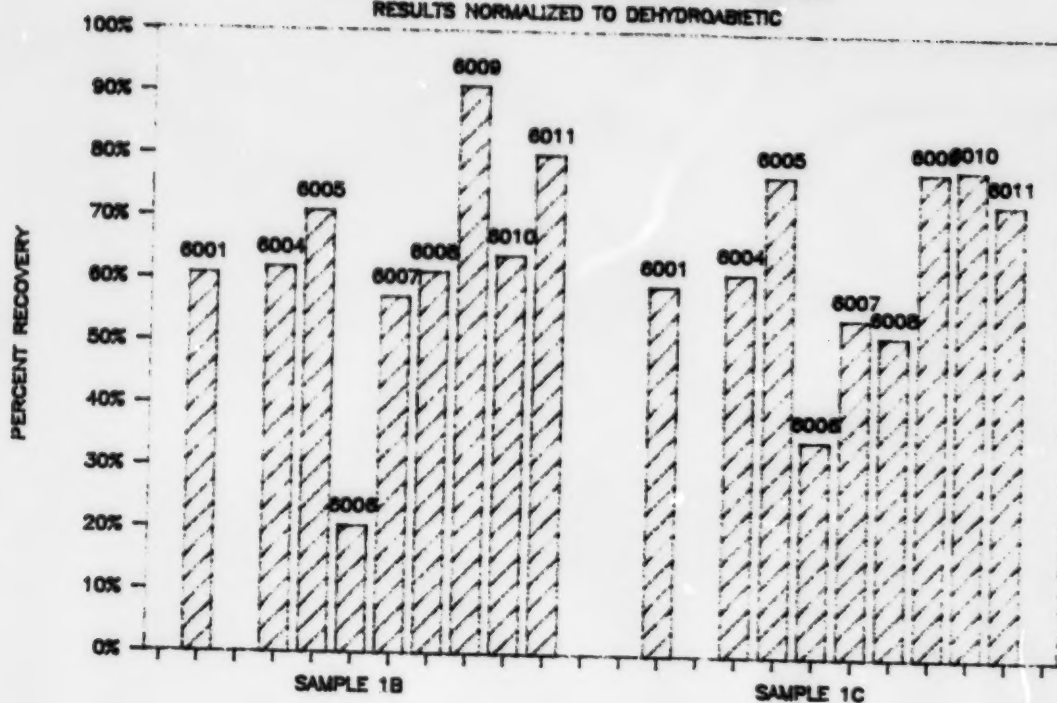
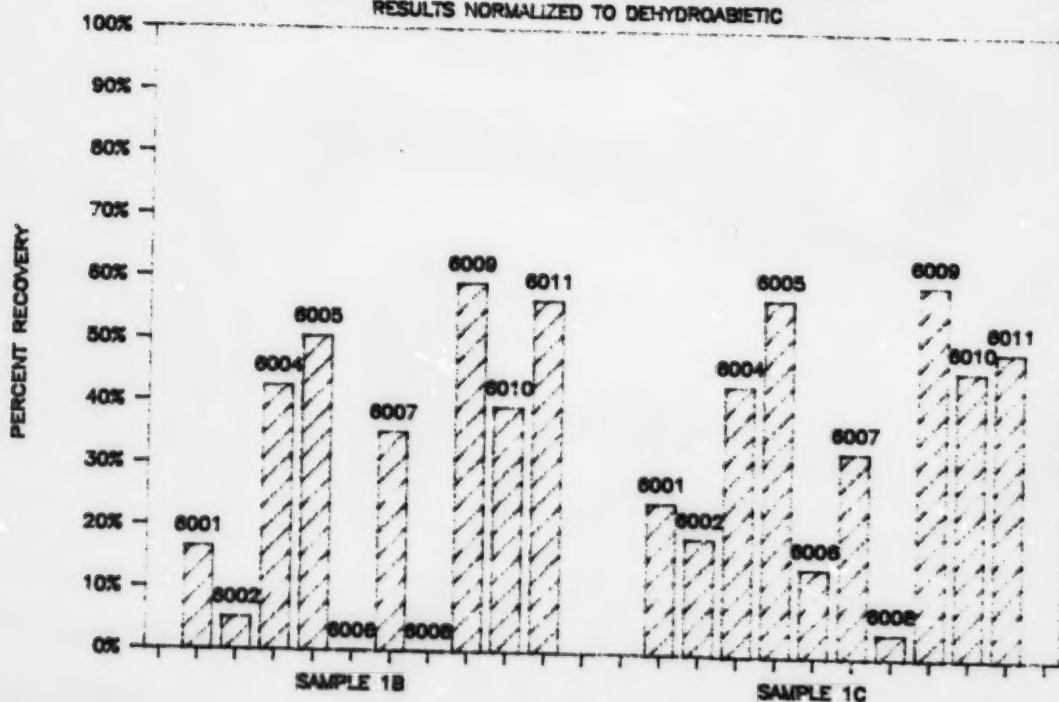


FIGURE 12: NEOABIETIC ACID

RESULTS NORMALIZED TO DEHYDROABIETIC



1

2

FIGURE 13: CHLORODEHYDROABIETIC ACID

RESULTS NORMALIZED TO DEHYDROABIETIC

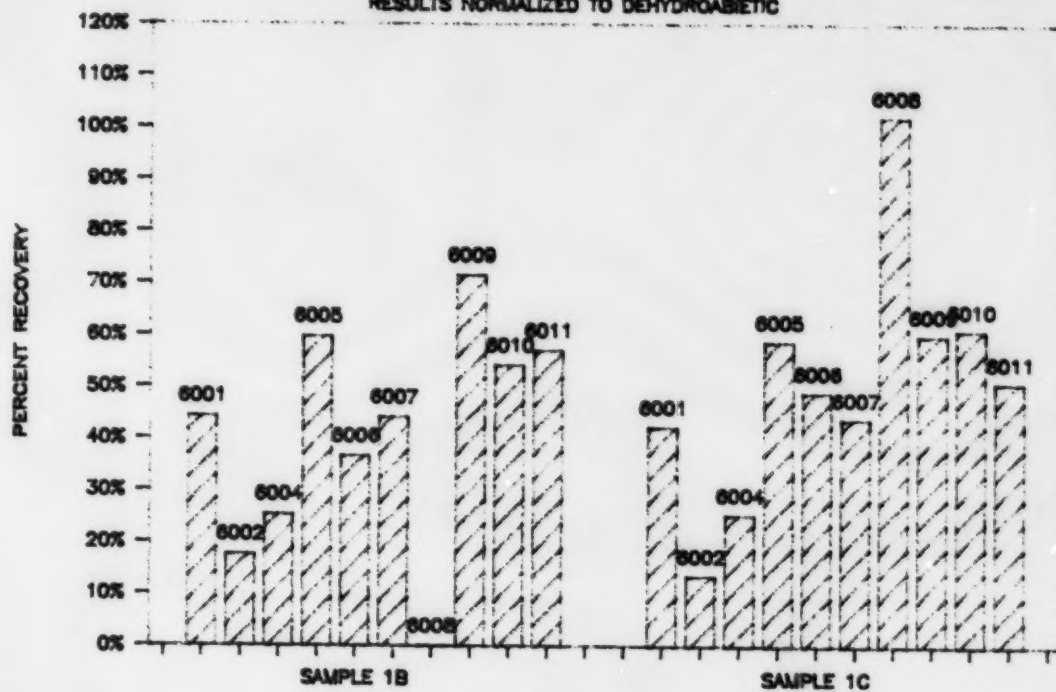


FIGURE 14: 9,10-DICHLOROSTEARIC ACID

RESULTS NORMALIZED TO DEHYDROABIETIC

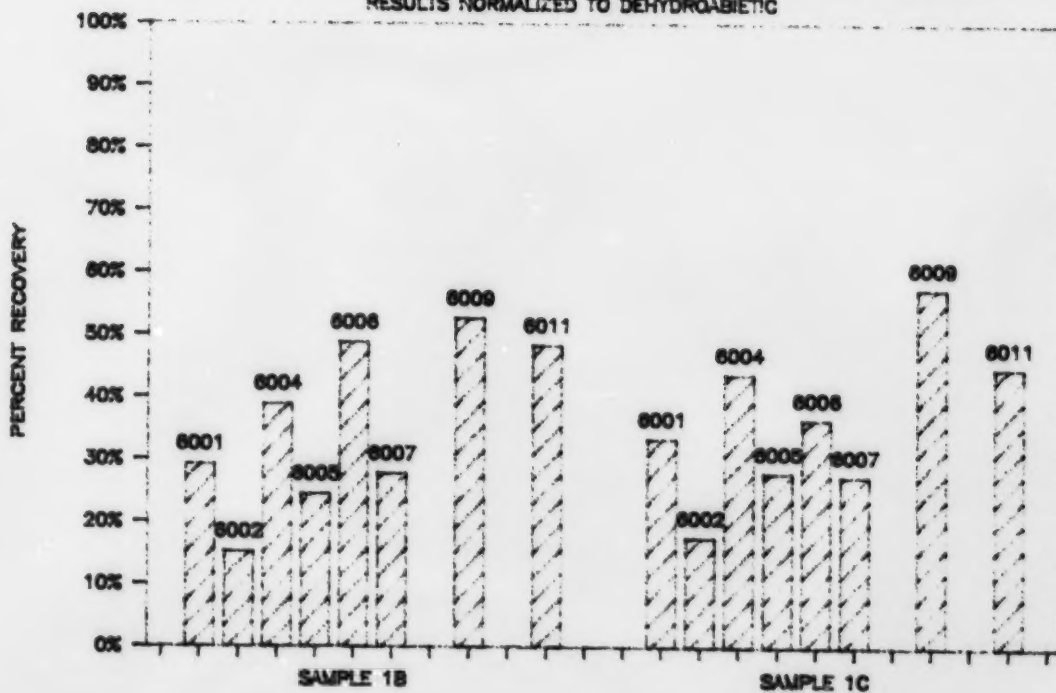
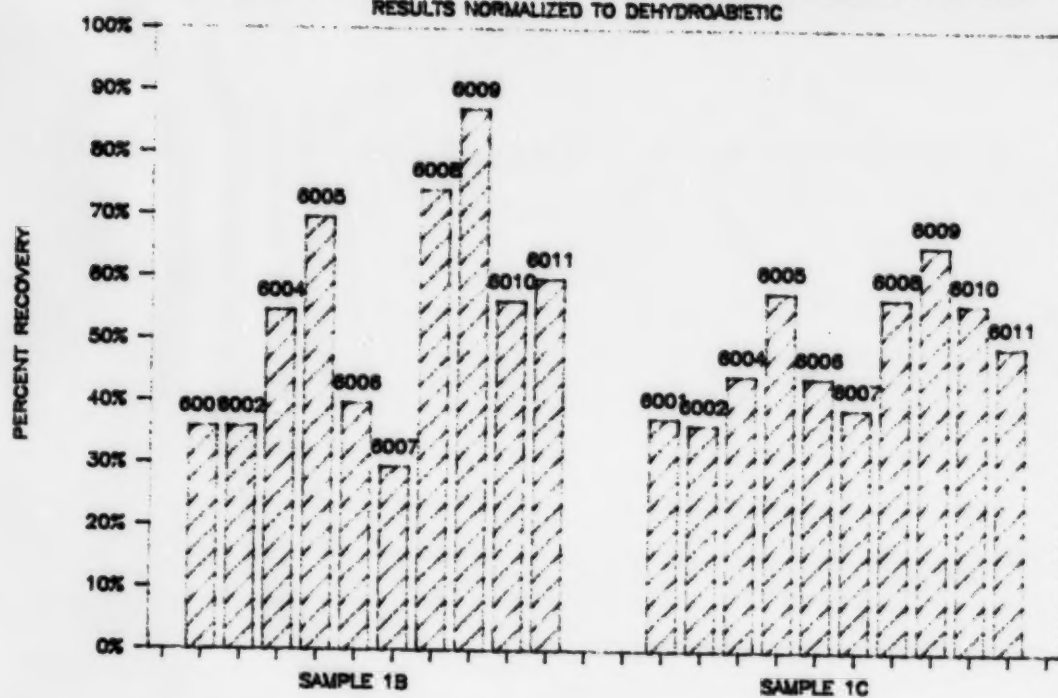


FIGURE 15: DICHLORODEHYDROABIETIC ACID
RESULTS NORMALIZED TO DEHYDROABIETIC





8 APPENDIX 2 - LIST OF PARTICIPANTS AND CORRESPONDENCE

List of Participants

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(514) 630-4100

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Contact: Dr. Medhat Riskala

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Contact: Dr. Jim McKinley

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Contact: Mr. Peter Henniger

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Contact: Mrs. Glee Burch

Ontario Ministry of the Environment
Laboratory Services Branch
Trace Organics Section
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Contact: Mrs. Yvonne Jones

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Contact: Ms. Donna McCurvin

RESIN AND FATTY ACID ROUND ROBIN NOTIFICATION
MOE 89-2: February 27, 1989

INTRODUCTION

This document is a formal notification of the Resin and Fatty Acid Method Development Round Robin, MOE 89-2, being conducted by the Ontario Ministry of the Environment on behalf of the MISA Pulp and Paper Analytical Working Group. It is hoped that full participation from the invited laboratories may occur.

METHODOLOGY

The draft method dated May, 1988 is to be used for MOE 89-2. Any participating laboratories who require an additional copy of the draft method should contact Sylvia Cussion at (416) 235-5842 or Steve Burns at (416) 235-5932.

Please note that the MISA working group requests that the method be followed as closely as possible. Any variations should be reported with the results. The MISA working group also requests that copies of the sample chromatograms be included with the final results.

SCHEDULE

During the week of March 13, 1989, participating laboratories will receive nine (9) spiked samples containing the following resin acids:

Sandaracopimaric Acid	Isopimaric Acid
Palustric Acid	Levopimaric Acid
Dehydroabietic Acid	Abietic Acid
Neoabietic Acid	Chlorodehydroabietic Acid
Dichlorodehydroabietic Acid	Oleic Acid
9,10-Dichlorostearic Acid	

All samples are to be extracted and analyzed according to the procedures in the draft method. The samples will consist of high and low spikes, plus blanks in distilled water and two different pulp and paper effluents.

Samples must be stored at 4 degrees Celcius upon receipt. Samples must be extracted within seven (7) days. The pH should not be adjusted until just prior to extraction (this differs slightly from section 9.2 of the draft method). Analysis of the extract should take place as soon after extraction as possible. Results must be reported no later than April 13, 1989 to Sylvia Cussion at the following address:

Ministry of the Environment
Laboratory Services Branch
Computer Systems - QA/QC Section
125 Resources Rd.
Rexdale, Ontario, M9W 5L1

Results received after April 13, 1989 will not be included in the final report.

SUMMARY OF RESULTS

All participating laboratories will be assigned an identification code. All laboratories will receive a complete set of the results from MOE 89-2. All laboratories will be identified only by their identification code. Conclusions drawn by the Analytical Working Group and recommendations made will also be provided to the participating laboratories. A final report including the results from the two Resin and Fatty Acid Round Robins conducted in 1988 by the MOE will be written in the summer of 1989.

No further notice is to be given prior to distribution of the samples. Should a laboratory wish to withdraw from this round robin, or if there are any difficulties with the above schedule, please contact Sylvia Cussion immediately at (416) 235-5842.

Ontario Ministry of the Environment
Laboratory Services Branch
LCS-QA/QC Section
125 Resources Rd.
Rexdale, Ontario, M9W 5L1
(416) 235-5842
FAX (416) 235-5744

March 21, 1989.

TO: PARTICIPANTS OF MOE RFA ROUND ROBIN 89-2

Please find enclosed nine (9) 1 litre amber bottles labelled 1A, 1B, 1C, 2A, 2B, 2C, 3A, 3B, and 3C. If you are missing any of the above items, please contact me at the above phone number immediately. Store all samples at 4 degrees Celcius until ready for extraction. Do not adjust the pH until just prior to extraction.

Samples labelled "B" and "C" have been spiked with the following compounds:

Sandaracopimaric Acid	Isopimaric Acid
Palustric Acid	Levopimaric Acid
Dehydroabietic Acid	Neoabietic Acid
Abietic Acid	Oleic Acid
Chlorodehydroabietic Acid	9,10-Dichlorostearic Acid
Dichlorodehydroabietic Acid	

These samples are to be extracted within seven (7) days of reception and analyzed as soon as possible thereafter. The results must be reported to me no later than April 21, 1989. Results received after this date will not be included in the final report. Results transmitted by FAX by April 21 are acceptable.

Please report all results as $\mu\text{g/L}$ (ppb). To aid the MISA working group's interpretation of the results, please include a copy of the sample chromatograms, your response factor and calculations for Dehydroabietic Acid, and any variations used from the Draft Method. Please report any problems with the analysis of the samples.

Your lab identification code is:

Please contact me if there are any questions.

Sincerely,

Sylvia Cussion
Laboratory Quality Audit Scientist

Ontario Ministry of the Environment
Laboratory Services Branch
LCS-QA/QC Section
125 Resources Rd.
Rexdale, Ontario
M9W 5L1

(416) 235-5842

June 6, 1989.

TO: PARTICIPANTS OF MOE INTERLABORATORY STUDY 89-2

Thank you for your participation in the MOE Inter-Laboratory Study 89-2 conducted in March 1989 on behalf of the Pulp and Paper Sector MISA Analytical Working Group. Your participation is greatly appreciated by the MISA Analytical Working Group.

Attached are all the results reported to me as of June 5, 1989. Not all the original participants were able to report results. Listed for each sample and parameter are the expected values, the results reported by each participant, the mean, minimum result, maximum result, and the standard deviation. For labs that did not report a result for a specific parameter for all samples, the space has been left blank. For labs that reported results for a parameter, but did not report a result in a particular sample, the result was reported as "0". All participants are identified only by their identification code.

A formal report is presently being written, but I regret that it will not be completed until August 1989. All participants will receive a copy as soon as it becomes available.

Please contact me if you have any further questions.

Sincerely,

Sylvia Cussion
Laboratory Quality Audit Scientist

Attachment

**9 APPENDIX 3 - ADDITIONAL DATA SETS USING VARIATIONS FROM
THE DRAFT METHOD**

Table 3 **Additional Results Using GC/MS**

Table 4 **Additional Results Using Post-Methylation Selective Clean-Up**

TABLE 3 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
 ADDITIONAL RESULTS USING GC/MS
 RESULTS EXPRESSED IN ppb AND AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: MARCH 21, 1989		ORIGINAL (06/02/89)				REVISED (07/28/89)					
SAMPLE#	PARAMETER	DESIGN	6002	6002	6002	6002	6002	6010	6010	RES	%REC
		(ppb)	RES1	RES2	%REC1	%REC2	RES1	RES2	%REC1	%REC2	
1A	OLEIC	-	80				20				
	SANDAROCOPIMARIC	-									
	ISOPIMARIC	-	14				14				
	PALUSTRIC/LEVOPIMARIC	-	-				-				
	PALUSTRIC	-	76				19				
	LEVOPIMARIC	-	230				58				
	DEHYDROABIETIC	-									
	ABIETIC	-									
	NEOABIETIC	-									
	CHLORODEHYDROABIETIC	-									
	9,10-DICHLOROSTEARIC	-	84				21				
	DICHLORODEHYDROABIETIC	-	64				16				
	o-METHYLPODOCARPIC ACID (%)		104.0%				104.0%				91.7%
1B	OLEIC	50	230	210	460%	420%	57	52	114%	104%	53.6 107%
	SANDAROCOPIMARIC	44.7	290	300	649%	671%	73	75	163%	168%	69.7 156%
	ISOPIMARIC	54.5	400	440	734%	807%	100	110	183%	202%	90.6 166%
	PALUSTRIC/LEVOPIMARIC	94.6									123.4 130%
	PALUSTRIC	44.6	270	290	605%	650%	68	73	152%	164%	
	LEVOPIMARIC	50	110	120	220%	240%	28	31	56%	62%	
	DEHYDROABIETIC	50	560	560	1120%	1120%	140	140	280%	280%	86.4 173%
	ABIETIC	47.2	840	920	1780%	1949%	210	230	445%	487%	105.9 224%
	NEOABIETIC	50	220	210	440%	420%	54	53	108%	106%	75 150%
	CHLORODEHYDROABIETIC	47.3	390	400	825%	846%	97	100	205%	211%	91.3 193%
	9,10-DICHLOROSTEARIC	48	320	350	667%	729%	80	87	167%	181%	
	DICHLORODEHYDROABIETIC	50	320	350	640%	700%	80	88	160%	176%	88.6 177%
	o-METHYLPODOCARPIC ACID (%)		101.0%	105.0%			101.0%	105.0%			96.8%
	MEAN RESULTS WITHIN SCAN		359.1	377.3			89.7	94.5			87.2
1C	OLEIC	400	800		200%		200		50%		227.5 57%
	SANDAROCOPIMARIC	357.6	1000		280%		260		73%		288.8 81%
	ISOPIMARIC	436	1600		367%		400		92%		376.7 86%
	PALUSTRIC/LEVOPIMARIC	756.8									503.4 67%
	PALUSTRIC	356.8	1200		336%		290		81%		
	LEVOPIMARIC	400	560		140%		140		35%		
	DEHYDROABIETIC	400	2100		525%		520		130%		371.4 93%
	ABIETIC	377.6	4000		1059%		1000		265%		426.6 113%
	NEOABIETIC	400	880		220%		220		55%		313.3 78%
	CHLORODEHYDROABIETIC	378.4	1500		396%		380		100%		335.3 89%
	9,10-DICHLOROSTEARIC	384	1200		313%		310		81%		
	DICHLORODEHYDROABIETIC	400	1400		350%		340		85%		286.8 72%
	o-METHYLPODOCARPIC ACID (%)		105.0%				105.0%				100.5%
	MEAN RESULTS WITHIN SCAN		1476.4				369.1				347.8

LABORATORY 6002 REISSUED ITS RESULTS AFTER INITIAL DISTRIBUTION OF RESULTS ON JUNE 5, 1989

o-METHYLPODOCARPIC ACID/TRICOSANOIC ACID - SURROGATE STANDARDS

REAGENT WATER: 1A - UNSPIKED; 1B - LOW SPIKE; 1C - HIGH SPIKE

TABLE 3 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
 ADDITIONAL RESULTS USING GC/MS
 RESULTS EXPRESSED IN PPB AND AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: MARCH 21, 1989		ORIGINAL (06/02/89)				REVISED (07/28/89)						
SAMPLE#	PARAMETER	DESIGN	6002		6002		6002		6002		6010	
		(ppb)	RES1	RES2	%REC1	%REC2	RES1	RES2	%REC1	%REC2	RES	%REC
2A	OLEIC	-	130				33					
	SANDAROCOPIMARIC	-										
	ISOPIMARIC	-	60				15					
	PALUSTRIC/LEVOPIMARIC	-	-									
	PALUSTRIC	-	68				17					
	LEVOPIMARIC	-	230				58					
	DEHYDROABIETIC	-										
	ABIETIC	-	48				12					
	NEOABIETIC	-										
	CHLORODEHYDROABIETIC	-										
	9,10-DICHLOROSTEARIC	-	100				25					
	DICHLORODEHYDROABIETIC	-										
o-METHYLPODOCARPIC ACID (%)			102.0%				102.0%				105.7%	
2B	OLEIC	50	120	120	240%	240%	29	29	58%	58%	0	0%
	SANDAROCOPIMARIC	44.7	0	0	0%	0%	0	0	0%	0%	11.1	25%
	ISOPIMARIC	54.5	0	0	0%	0%	0	0	0%	0%	13.2	24%
	PALUSTRIC/LEVOPIMARIC	94.6									22.2	23%
	PALUSTRIC	44.6	0	0	0%	0%	0	0	0%	0%		
	LEVOPIMARIC	50	220	224	440%	448%	55	49	110%	98%		
	DEHYDROABIETIC	50	40	40	80%	80%	10	10	20%	20%	39.9	80%
	ABIETIC	47.2	92	76	195%	161%	23	19	49%	40%	34	72%
	NEOABIETIC	50	0	0	0%	0%	0	0	0%	0%	21.5	43%
	CHLORODEHYDROABIETIC	47.3	144	144	304%	304%	36	36	76%	76%	44	93%
	9,10-DICHLOROSTEARIC	48	120	110	250%	229%	29	27	60%	56%		
	DICHLORODEHYDROABIETIC	50	210	210	420%	420%	53	52	106%	104%	0	0%
o-METHYLPODOCARPIC ACID (%)			103.0%	105.0%			103.0%	105.0%			95.0%	
MEAN RESULTS WITHIN SCAN			86.0	84.0			21.4	20.2			20.7	
2C	OLEIC	400	150		38%		37		9%		0	0%
	SANDAROCOPIMARIC	357.6	640		179%		160		45%		193.4	54%
	ISOPIMARIC	436	1040		239%		260		60%		248	57%
	PALUSTRIC/LEVOPIMARIC	756.8									268.1	35%
	PALUSTRIC	356.8	310		87%		77		22%			
	LEVOPIMARIC	400	56		14%		14		4%			
	DEHYDROABIETIC	400	1000		250%		250		63%		267.4	67%
	ABIETIC	377.6	2200		583%		550		146%		295.9	78%
	NEOABIETIC	400	480		120%		120		30%		216.9	54%
	CHLORODEHYDROABIETIC	378.4	1200		317%		290		77%		390.5	103%
	9,10-DICHLOROSTEARIC	384	280		73%		70		18%			
	DICHLORODEHYDROABIETIC	400	1200		300%		300		75%		295.6	74%
o-METHYLPODOCARPIC ACID (%)			108.0%				108.0%				104.0%	
MEAN RESULTS WITHIN SCAN			777.8				193.5				241.8	

LABORATORY 6002 REISSUED ITS RESULTS AFTER INITIAL DISTRIBUTION OF RESULTS ON JUNE 5, 1989

o-METHYLPODOCARPIC ACID/TRICOSANOIC ACID - SURROGATE STANDARDS

PULP AND PAPER EFFLUENT #1: 2A - UNSPIKED; 2B - LOW SPIKE; 2C - HIGH SPIKE

TABLE 3 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS

ADDITIONAL RESULTS USING GC/MS

RESULTS EXPRESSED IN ppb AND AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: MARCH 21, 1989

DISTRIBUTED: MARCH 21, 1989		ORIGINAL (06/02/89)				REVISED (07/28/89)						
SAMPLE#	PARAMETER	DESIGN	6002		6002		6002		6002		6010	
		(ppb)	RES1	RES2	%REC1	%REC2	RES1	RES2	%REC1	%REC2	RES	%REC
3A	OLEIC	-	230				29				2	
	SANDAROCOPIMARIC	-									2	
	ISOPIMARIC	-										
	PALUSTRIC/LEVOPIMARIC	-	-				-					
	PALUSTRIC	-										
	LEVOPIMARIC	-	170				42					
	DEHYDROABIETIC	-	56				14					
	ABIETIC	-	120				29				8	
	NEOABIETIC	-										
	CHLORODEHYDROABIETIC	-										
	9,10-DICHLOROSTEARIC	-	100				26					
	DICHLORODEHYDROABIETIC	-									4	
	o-METHYLPDOCARPIC ACID (%)		98.0%				98.0%				92.8%	
3B	OLEIC	50	180		360%		44		88%		21.7	43%
	SANDAROCOPIMARIC	44.7	0		0%		0		0%		0	0%
	ISOPIMARIC	54.5	0		0%		0		0%		2.6	5%
	PALUSTRIC/LEVOPIMARIC	94.6									0	0%
	PALUSTRIC	44.6	0		0%		0		0%			
	LEVOPIMARIC	50	200		400%		51		102%			
	DEHYDROABIETIC	50	64		128%		16		32%		0	0%
	ABIETIC	47.2	140		297%		36		76%		9.4	20%
	NEOABIETIC	50	0		0%		0		0%		0	0%
	CHLORODEHYDROABIETIC	47.3	40		85%		0		0%		0	0%
	9,10-DICHLOROSTEARIC	48	160		333%		40		83%			
	DICHLORODEHYDROABIETIC	50	100		200%		26		52%		16.7	33%
	o-METHYLPDOCARPIC ACID (%)		105.0%				105.0%				108.0%	
MEAN RESULTS WITHIN SCAN			80.4				19.4				5.6	
3C	OLEIC	400	140	170	35%	43%	36	42	9%	11%	4.7	1%
	SANDAROCOPIMARIC	357.6	0	0	0%	0%	0	0	0%	0%	2.5	1%
	ISOPIMARIC	436	52	64	12%	15%	13	16	3%	4%	5.4	1%
	PALUSTRIC/LEVOPIMARIC	756.8									3.6	0%
	PALUSTRIC	356.8	0	0	0%	0%	0	0	0%	0%		
	LEVOPIMARIC	400	190	220	48%	55%	47	55	12%	14%		
	DEHYDROABIETIC	400	68	76	17%	19%	17	19	4%	5%	8.1	2%
	ABIETIC	377.6	180	170	48%	45%	44	43	12%	11%	12.2	3%
	NEOABIETIC	400	0	0	0%	0%	0	0	0%	0%	0	0%
	CHLORODEHYDROABIETIC	378.4	200	210	53%	55%	26	27	7%	7%	28.2	7%
	9,10-DICHLOROSTEARIC	384	120	130	31%	34%	30	33	8%	9%		
	DICHLORODEHYDROABIETIC	400	370	390	93%	98%	93	97	23%	24%	113.3	28%
	o-METHYLPDOCARPIC ACID (%)		99.0%	106.0%			99.0%	106.0%			85.0%	
MEAN RESULTS WITHIN SCAN			120.0	130.0			27.8	30.2			19.8	

LABORATORY 6002 REISSUED ITS RESULTS AFTER INITIAL DISTRIBUTION OF RESULTS ON JUNE 5, 1989

o-METHYLPDOCARPIC ACID/TRICOSANOIC ACID - SURROGATE STANDARDS

PULP AND PAPER EFFLUENT #2: 3A - UNSPIKED; 3B - LOW SPIKE; 3C - HIGH SPIKE

TABLE 4 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
 ADDITIONAL RESULTS USING POST-METHYLATION SELECTIVE CLEAN-UP
 RESULTS EXPRESSED IN ppb AND AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: MARCH 21, 1989

SAMPLE#	PARAMETER	DESIGN	RES1	RES2	%REC1	%REC2
		(ppb)				
1A	OLEIC	-	25	32		
	SANDAROCOPIMARIC	-				
	ISOPIMARIC	-				
	PALUSTRIC	-				
	LEVOPIMARIC	-				
	DEHYDROABIETIC	-				
	ABIETIC	-	11	25		
	NEOABIETIC	-	9	12		
	CHLORODEHYDROABIETIC **	-				
	CHLORODEHYDROABIETIC **	-				
	9,10-DICHLOROSTEARIC	-				
	DICHLORODEHYDROABIETIC	-				
	* PALMITIC		43	42		
	* LINOLEIC		94	134		
	* LINOLENIC		70	122		
	* STEARIC		54	62		
	* PIMARIC					
1B	OLEIC	50	84	45	168%	90%
	SANDAROCOPIMARIC	44.7	95	101	213%	226%
	ISOPIMARIC	54.5	112	128	206%	235%
	PALUSTRIC	44.6	109	114	244%	256%
	LEVOPIMARIC	50	147	14	294%	28%
	DEHYDROABIETIC	50	176	206	352%	412%
	ABIETIC	47.2	129	151	273%	320%
	NEOABIETIC	50	111	117	222%	234%
	CHLORODEHYDROABIETIC **	47.3	155	194	600%	899%
	CHLORODEHYDROABIETIC **		129	231		
	9,10-DICHLOROSTEARIC	48	146	116	304%	242%
	DICHLORODEHYDROABIETIC	50	209	53	418%	106%
	* PALMITIC		44	43		
	* LINOLEIC		157	65		
	* LINOLENIC		98	127		
	* STEARIC		62	33		
	* PIMARIC		8	10		
MEAN RESULTS WITHIN SCAN			133.5	122.5		

* ADDITIONAL PARAMETERS NOT IN THE SPIKING MATERIAL
 ** RESULTS FOR BOTH ISOMERS REPORTED SEPARATELY; EXPECTED VALUE &
 PERCENT RECOVERY CALCULATED FOR BOTH ISOMERS COMBINED.
 REAGENT WATER: 1A - UNSPIKED; 1B - LOW SPIKE; 1C - HIGH SPIKE

TABLE 4 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
 ADDITIONAL RESULTS USING POST-METHYLATION SELECTIVE CLEAN-UP
 RESULTS EXPRESSED IN ppb AND AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: MARCH 21, 1989

SAMPLE#	PARAMETER	DESIGN (ppb)	RES1	RES2	%REC1	%REC2
1C	OLEIC	400	155	179	39%	45%
	SANDAROCOPIMARIC	357.6	229	242	64%	68%
	ISOPIMARIC	436	293	299	67%	69%
	PALUSTRIC	356.8	242	271	68%	76%
	LEVOPIMARIC	400	340	367	85%	92%
	DEHYDROABIETIC	400	348	349	87%	87%
	ABIETIC	377.6	220	239	58%	63%
	NEOABIETIC	400	230	246	58%	62%
	CHLORODEHYDROABIETIC **	378.4	207	222	101%	108%
	CHLORODEHYDROABIETIC **		174	187		
	9,10-DICHLOROSTEARIC	384	363	398	95%	104%
	DICHLORODEHYDROABIETIC	400	271	290	68%	73%
	* PALMITIC		14	14		
	* LINOLEIC		28	31		
	* LINOLENIC		20	16		
	* STEARIC		15	13		
	* PIMARIC		9	9		
	MEAN RESULTS WITHIN SCAN		256.0	274.1		

* ADDITIONAL PARAMETERS NOT IN THE SPIKING MATERIAL

** RESULTS FOR BOTH ISOMERS REPORTED SEPARATELY; EXPECTED VALUE &
 PERCENT RECOVERY CALCULATED FOR BOTH ISOMERS COMBINED.

REAGENT WATER: 1A - UNSPIKED; 1B - LOW SPIKE; 1C - HIGH SPIKE

TABLE 4 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
 ADDITIONAL RESULTS USING POST-METHYLATION SELECTIVE CLEAN-UP
 RESULTS EXPRESSED IN ppb AND AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: MARCH 21, 1989

SAMPLE#	PARAMETER	DESIGN (ppb)	RES1	RES2	%REC1	%REC2
2A	OLEIC	-	6	5		
	SANDAROCOPIMARIC	-				
	ISOPIMARIC	-				
	PALUSTRIC	-				
	LEVOPIMARIC	-				
	DEHYDROABIETIC	-				
	ABIETIC	-				
	NEOABIETIC	-	14	14		
	CHLORODEHYDROABIETIC **	-		7		
	CHLORODEHYDROABIETIC **	-	17	13		
	9,10-DICHLOROSTEARIC	-	16	13		
	DICHLORODEHYDROABIETIC	-	5			
	* PALMITIC		6	13		
	* LINOLEIC					
	* LINOLENIC		12	21		
	* STEARIC		5	10		
	* PIMARIC		5			
2B	OLEIC	50	0	14	0%	28%
	SANDAROCOPIMARIC	44.7	15	17	34%	38%
	ISOPIMARIC	54.5	15	0	28%	0%
	PALUSTRIC	44.6	6	0	13%	0%
	LEVOPIMARIC	50	28	17	56%	34%
	DEHYDROABIETIC	50	6	0	12%	0%
	ABIETIC	47.2	14	29	30%	61%
	NEOABIETIC	50	11	17	22%	34%
	CHLORODEHYDROABIETIC **	47.3	22	31	47%	66%
	CHLORODEHYDROABIETIC **		0	0		
	9,10-DICHLOROSTEARIC	48	0	0	0%	0%
	DICHLORODEHYDROABIETIC	50	43	15	86%	30%
	* PALMITIC		13	15		
	* LINOLEIC		16	23		
	* LINOLENIC		28	18		
	* STEARIC		18	23		
	* PIMARIC					
MEAN RESULTS WITHIN SCAN			13.3	11.7		

* ADDITIONAL PARAMETERS NOT IN THE SPIKING MATERIAL

** RESULTS FOR BOTH ISOMERS REPORTED SEPARATELY; EXPECTED VALUE &
 PERCENT RECOVERY CALCULATED FOR BOTH ISOMERS COMBINED.

PULP AND PAPER EFFLUENT #1: 2A - UNSPIKED; 2B - LOW SPIKE; 2C - HIGH SPIKE

TABLE 4 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
 ADDITIONAL RESULTS USING POST-METHYLATION SELECTIVE CLEAN-UP
 RESULTS EXPRESSED IN ppb AND AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: MARCH 21, 1989

SAMPLE#	PARAMETER	DESIGN (ppb)	RES1	RES2	%REC1	%REC2
2C	OLEIC	400	16	9	4%	2%
	SANDAROCOPIMARIC	357.6	7	0	2%	0%
	ISOPIMARIC	436	7	0	2%	0%
	PALUSTRIC	356.8	257	210	72%	59%
	LEVOPIMARIC	400	0	0	0%	0%
	DEHYDROABIETIC	400	318	252	80%	63%
	ABIETIC	377.6	101	74	27%	20%
	NEOABIETIC	400	168	156	42%	39%
	CHLORODEHYDROABIETIC **	378.4	221	168	164%	112%
	CHLORODEHYDROABIETIC **		401	257		
	9,10-DICHLOROSTEARIC	384	33	34	9%	9%
	DICHLORODEHYDROABIETIC	400	174	132	44%	33%
	* PALMITIC		6	7		
	* LINOLEIC			9		
	* LINOLENIC		25	16		
	* STEARIC			18		
	* PIMARIC		8	6		
	MEAN RESULTS WITHIN SCAN		141.9			

* ADDITIONAL PARAMETERS NOT IN THE SPIKING MATERIAL

** RESULTS FOR BOTH ISOMERS REPORTED SEPARATELY; EXPECTED VALUE &
 PERCENT RECOVERY CALCULATED FOR BOTH ISOMERS COMBINED.

PULP AND PAPER EFFLUENT #1: 2A - UNSPIKED; 2B - LOW SPIKE; 2C - HIGH SPIKE

TABLE 4 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
 ADDITIONAL RESULTS USING POST-METHYLATION SELECTIVE CLEAN-UP
 RESULTS EXPRESSED IN ppb AND AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: MARCH 21, 1989

SAMPLE#	PARAMETER	DESIGN	RES1	RES2	%REC1	%REC2
		(ppb)				
3A	OLEIC	-	21	17		
	SANDAROCOPIMARIC	-		11		
	ISOPIMARIC	-				
	PALUSTRIC	-	7	17		
	LEVOPIMARIC	-				
	DEHYDROABIETIC	-	6	8		
	ABIETIC	-	6	8		
	NEOABIETIC	-	21	26		
	CHLORODEHYDROABIETIC **	-	17	31		
	CHLORODEHYDROABIETIC **	-	8	47		
	9,10-DICHLOROSTEARIC	-		28		
	DICHLORODEHYDROABIETIC	-				
	* PALMITIC		17	23		
	* LINOLEIC		27	26		
	* LINOLENIC		20	5		
	* STEARIC		10	10		
	* PIMARIC		5	14		
3B	OLEIC	50	5	6	10%	12%
	SANDAROCOPIMARIC	44.7	5	0	11%	0%
	ISOPIMARIC	54.5	0	0	0%	0%
	PALUSTRIC	44.6	5	7	11%	16%
	LEVOPIMARIC	50	16	17	32%	34%
	DEHYDROABIETIC	50	5	7	10%	14%
	ABIETIC	47.2	6	7	13%	15%
	NEOABIETIC	50	23	24	46%	48%
	CHLORODEHYDROABIETIC **	47.3	0	0	0%	15%
	CHLORODEHYDROABIETIC **		0	7		
	9,10-DICHLOROSTEARIC	48	5	4	10%	8%
	DICHLORODEHYDROABIETIC	50	14	16	28%	32%
	* PALMITIC		18	23		
	* LINOLEIC		37	40		
	* LINOLENIC		19	28		
	* STEARIC		12	15		
	* PIMARIC			6		
MEAN RESULTS WITHIN SCAN			7.0	7.9		

* ADDITIONAL PARAMETERS NOT IN THE SPIKING MATERIAL

** RESULTS FOR BOTH ISOMERS REPORTED SEPARATELY; EXPECTED VALUE &
 PERCENT RECOVERY CALCULATED FOR BOTH ISOMERS COMBINED.

PULP AND PAPER EFFLUENT #2: 3A - UNSPIKED; 3B - LOW SPIKE; 3C - HIGH SPIKE

TABLE 4 - INTERLABORATORY STUDY 89-2: RESIN AND FATTY ACIDS
 ADDITIONAL RESULTS USING POST-METHYLATION SELECTIVE CLEAN-UP
 RESULTS EXPRESSED IN ppb AND AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: MARCH 21, 1989

SAMPLE#	PARAMETER	DESIGN	RES1	RES2	%REC1	%REC2
		(ppb)				
3C	OLEIC	400	6	6	2%	2%
	SANDAROCOPIIMARIC	357.6	6	0	2%	0%
	ISOPIMARIC	436	21	0	5%	0%
	PALUSTRIC	356.8	10	8	3%	2%
	LEVOPIIMARIC	400	24	16	6%	4%
	DEHYDROABIETIC	400	15	6	4%	2%
	ABIETIC	377.6	10	8	3%	2%
	NEOABIETIC	400	26	23	7%	6%
	CHLORODEHYDROABIETIC **	378.4	7	7	5%	5%
	CHLORODEHYDROABIETIC **		12	11		
	9,10-DICHLOROSTEARIC	384	9	5	2%	1%
	DICHLORODEHYDROABIETIC	400	78	80	20%	20%
	* PALMITIC		19	23		
	* LINOLEIC		40	39		
	* LINOLENIC		19	22		
	* STEARIC		14	12		
	* PIMARIC		7	5		
MEAN RESULTS WITHIN SCAN			18.7	14.2		

* ADDITIONAL PARAMETERS NOT IN THE SPIKING MATERIAL

** RESULTS FOR BOTH ISOMERS REPORTED SEPARATELY; EXPECTED VALUE &
 PERCENT RECOVERY CALCULATED FOR BOTH ISOMERS COMBINED.

PULP AND PAPER EFFLUENT #2: 3A - UNSPIKED; 3B - LOW SPIKE; 3C - HIGH SPIKE